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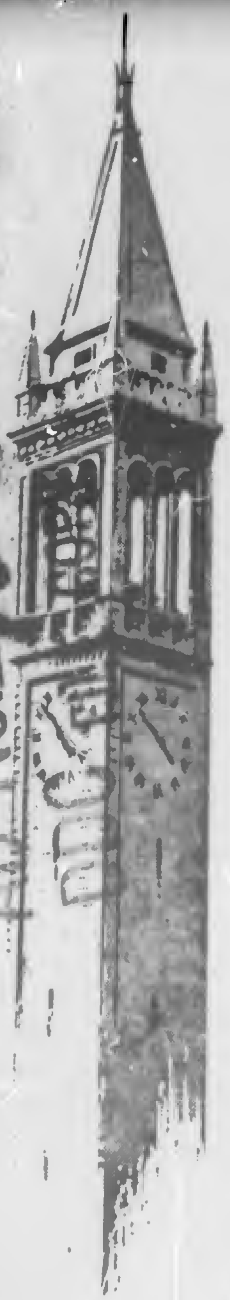

CRITICAL COMPILATION OF CERAMIC FORMING METHODS

Final Report

A. G. Pincus, project coordinator

Report No. MT-63-12
Contract AF 33(657)-10574
September 1963

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⑪ ~~September~~ 1963)

⑭ Rept. no. MT63-12

AF Materials Laboratory
Research and Technology Division
Air Force Systems Command
Wright-Patterson Air Force Base, Ohio

⑯ Proj~~ect No~~. 7381 ⑰ Task ~~No.~~: 738105

(Prepared under ⑮ Contract [illegible] AF 33(657)-10574
by the University of California, Berkeley, California;
College of Engineering, Department of Mineral Technology;
A. G. Pincus, project coordinator

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NOTICES

FOREWORD

(21) Bypart on

This report was prepared by the University of California, under USAF Contract No. AF 33(657)-10574. This contract was initiated under Project No. 7381, "Materials Application," Task No. 738105, "Ceramic and Graphite Technical Information". The project and report have been the responsibility of the Department of Mineral Technology, College of Engineering, University of California at Berkeley with Professor D. W. Fuerstenau as faculty investigator, A. G. Pincus as project coordinator, and an active staff of consultants and specialists as acknowledged on the following page. In addition more than 110 experts in the forming of ceramics volunteered advice to the project by attendance at intensive day-long conferences, private discussions, and submission of memos and literature. They represent an extensive cross-section of companies active in the forming of ceramics by the variety of techniques described in the report, as well as specialists from universities, government laboratories, and publications. It is regretted that they can not each be cited by name.

The work was administered under the direction of the AF Materials Laboratory, Research and Technology Division, with Mr. C. E. Butler as acting chief, Ceramics and Graphite Technical Evaluation section during the early stages of the contract and Mr. S. W. Bradstreet as chief starting May 15, 1963. Mr. Lawrence Kopell has been project engineer.

This report covers work done from April 1963 to September 1963.

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ACKNOWLEDGMENTS

The staff wishes to acknowledge the special help so graciously contributed by Mr. W. J. Tonkin and his entire office staff of the Institute of Engineering Research, University of California, Berkeley. Also Mr. John V. Kauffman, Contracting Officer, Aeronautical Systems Command, Wright-Patterson Air Force Base. Thanks are due to various members of the Inorganic Materials Research Division Lawrence Radiation Laboratory, University of California. Professor Pask and Dr. Pincus served on the Ad Hoc Committee on Processing of Ceramic Materials of the Materials Advisory Board, National Academy of Science/National Research Council from October 1962 to September 1963 and acknowledgements are made for the abundant information and ideas from the activities of the committee and from its report which have aided this project.

ABSTRACT

A first phase study is presented on methods for forming and characterizing ceramics. Five reviews by individual authors appraise the state of the art in characterizing the raw materials and batches and forming by cold, hot, melt, and miscellaneous processes. The derived fundamentals contributing to control over forming are briefly summarized. Initial bibliographies arranged or a specially evolved classification scheme introduce the literature on characterization of particles and assemblies of particles, and on forming of ceramics.

This technical documentary report has been reviewed and is approved.

D. A. SHINN
Chief, Materials Information Branch
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I. GENERAL INTRODUCTION

When the need arises for a specific design of a ceramic in a specific composition, a great number of competing methods are advanced for forming the shape required. The purpose of this study has been to initiate a critical study of the alternative forming methods in order to permit a selection based on reasoned judgments rather than on the grounds of familiarity, emotion, or traditions.

The report and the accompanying bibliographic and reference material provide a basis for the assembly of a uniform source of information on the forming of ceramic articles.

Forming must first be seen in perspective as one step in the total processing sequence for making ceramics comprising:

Batch Preparation
Forming
Heat Treatment
Finishing and Assembly
Inspection

Alternative terms for the forming operations include: shaping, molding, consolidation, and compaction.

The objective of processing as a whole is to produce ceramic or composite materials with a specified and reproducible characterization as to:

(1) chemical and mineral composition
(2) physical structure
(3) dimensions
(4) surface finish
(5) pertinent properties

The forming step plays an integral and significant part in achieving this objective.

The functions of forming are:

(1) To obtain a specified shape and size within dimensional tolerances yielding uniformity (This piece may be a blank, subject to further shaping and dimensional truing in the as-shaped, dried, partically fired, or fully matured stages.);

(2) To achieve a uniform composition and density throughout-the structure producing homogeneity;

(3) To contribute to a controlled microstructure and properties in the final produce promoting reproducibility.

Manuscript released by the authors September 1963 for publication as a RTD Technical Documentary Report

These functions are attained by consolidating discrete particles into a compact coherent enough to permit handling during further shaping or during setting for heat treatment. Implicit is the attainment during forming of:

(1) controlled density
(2) freedom from gross flaws
(3) minimized contamination
(4) safety with regard to such factors as toxicity, radioactivity, and die strengths.

A subsidiary function of consolidation may be to accelerate reaction to final composition and structure during subsequent heat treatment. The final surface finish may be established by the forming, or modified during a subsequent heat treatment or finishing stage.

Since throughout this survey ceramics are being defined to include all materials which are nonmetallic and inorganic (including composites), forming has been considered in its broadest sense to include not only the familiar powder techniques, but also forming from melts, vapors, and solid bodies of varying degrees of porosity and bond strengths. The concept of "powders" has been broadened to include all particulate matter whether equiaxed, filamentary or sheet-like.

Classifications of forming can be developed according to a diversity of approaches as follows.*

Sequential Operations.-- Feed material from the preparation stage in the form of a granulated powder batch, a preform, or a homogenized, controlled viscosity glass is (1) filled into a die cavity; and then (2) compacted, usually by pressure applied at a controlled rate. The formed part is then (3) ejected and (4) transported to the next operation.

During filling there are usually performed some auxiliary operations, such as removing excess feed material or de-airing. During compaction, the accompanying operations may include air release, lubrication of die walls, control of the pressure loading cycle, control of the degree of compaction, and multiple movements to form holes or re-entrants. Sometimes the simple application of the load by pressure is modified by introducing vibration, oscillation, or repetition of load.

The objective in each of these operations is maximum uniformity in structure throughout the compact, particularly with respect to density. Deviations at any stage may lead to more serious defects in subsequent processing.

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* This classification and discussion as well as other ideas embodied in other classifications have been derived from contributions of E. R. Stover to the Materials Advisory Board, Ad Hoc Committee on Processing of Ceramic Materials.

Method of Loading at Compact Boundaries. - The characteristics of the forming container are the basis of this classification: (1) elastic container, no shear-- corresponds to isostatic pressing, (2) rigid container, low shear-- corresponds to die pressing, (3) porous, rigid container, no shear-- slip casting, (4) powder is its own container, low shear-- powder rolling and strip compaction, (5) rigid container, high shear-- extrusion.

Operational Technique. - General operations or processes can be listed as: (1) pressing (compression), (2) drawing (tension), (3) gas pressure (blowing), (4) vibrating, (5) impacting, (6) leaching, (7) impregnating (8) weaving, (9) felting, (10) turning, (11) grinding, (12) casting and solidification, (13) chemical reactions (cement bonding, reaction sintering), (14) absorption (slip casting in porous molds). Combinations of these techniques may also be employed.

Material Response.- Previous classifications have emphasized operational features and effects on the material. Another method of classification might be based on the deformation response in the material during forming. This is discussed at length in the section of this report on Cold Forming Processes.

Type of Energy Utilized. - The method of energy utilization during forming may be listed as: (1) mechanical, (2) mechanical assisted by heat, (3) sonically assisted, (4) electrically assisted, (5) radiation induced, (6) magnetically assisted, (7) chemically-assisted, etc.

State of Matter from which Formed. - The states of matter from which ceramics are formed include: (1) particulates in varying solid-liquid ratios (actually a ternary: solid-air-liquid), (2) melts, (3) vapors, (4) bulk solids (including single crystals and glasses), (5) films, fibers, whiskers (i.e. solids with one or two dimensions dominating), (6) solutions.

Utilization. - The environment characteristic of a particular application may dictate properties required and the design of the component may specify the shape. Thus another basis for classification of forming methods may be defined by the end use of the ceramic. A typical example may be taken from the glass industry where the shape of the product determines the forming process (see table I of the section of this report on Melt Forming). Similar considerations have determined the evolution of forming processes in the whitewares industry for such products as dinnerware, bathtubs, and sparkplugs. Military requirements have begun to dictate the choice of forming methods for specific product categories like radomes, nose cones, rocket nozzles, and nuclear fuel elements. The influence of classification by this criterion is evident in the coding scheme and bibliography accompanying this report.

It is apparent from the diversity in scope of these classifications of forming methods that it is difficult to isolate and identify the science fundamentals underlying ceramic processing. However, two broad groups of fundamentals are suggested: those relating to the materials to be formed and those relating to the processing itself. If a true "science of processing" is to be generated, the detailed role of fundamentals in each of these groups and the relationships between the two groups must be discriminated and exploited. These concepts are introduced in the section of this report entitled Fundamentals of Forming, as well as being an integral part of the five individual survey reports which follow.

II. SURVEYS OF METHODS OF FORMING CERAMICS

For the purposes of this survey the forming processes have been broken down into the four arbitrary divisions:

Cold Forming
Hot Forming
Melt Forming
Miscellaneous Forming Methods.

Although any break down is bound to present borderline areas, the above groupings have been investigated without confusion both by discussions with experts and by a literature search. A more detailed classification of these groups of forming methods is presented in Section V.

Each of the four divisions were assigned to the responsible individual as set forth in the Table of Contents. These sought the information needed by means of individual and group conferences with recognized specialists in the several fields. Based on their findings and on the availability of the literature searched for their division, each of the leaders has prepared a summarizing report which is presented as a separate chapter substantially as written by them.

Because of this importance of the characterization of powders to subsequent forming operations was recognized from the beginning, and re-emphasized at all of the conferences, Professor D. W. Fuerstenau was asked to be responsible for the preparation of a special report and bibliography on this topic.

A. CHARACTERIZATION OF PARTICULATE SYSTEMS

by D. W. Fuerstenau and A. L. Mular

The basic idea behind the term particle characterization is simply that the engineer must accumulate a sufficient amount of data on the basic properties of the powder or particles which comprise a batch so as to be able to predict its behavior after a forming treatment. These same properties will be of consequence to the forming operation itself.

One forming operation may demand that a batch exhibit certain desirable properties which may not be of importance when another forming method is used. However, particles and assemblies of particles will always be characterized by such basic physical and chemical properties as size, color, shape, density, surface energy, and defect structure. The purpose of this section is to summarize the present state of knowledge regarding particle characterization and to show that properties which characterize batching materials pertain not only to forming but to most other subsequent operations.

PROPERTIES OF PARTICLES AND ASSEMBLIES OF PARTICLES

It is difficult to make a clear distinction between properties of a particle and properties of an assembly of particles. For example, a particle may be porous, yet when a pore size distribution is measured, it is done with an assembly of particles. Table I summarizes properties of particles which serve to determine their behavior in fluid environments. This table has served as the basis for a scheme of documentation entitled Particle Technology as noted in the Bibliography.

Table II presents a number of physico-chemical effects which are important in particle technology. The table lists the physico-chemical principle involved and some examples of its application. It is apparent that fundamental properties have a direct influence in a wide variety of areas which involve the use of powders. All of the principles listed in Table II may be regrouped and classified to fit into one of the general categories of Table I.

A considerable amount of work remains to determine those properties of particles which are uniquely related to specific forming methods, although in large part these properties will be important to all forming methods. Many of these properties which generally characterize particles are briefly discussed in the subsequent paragraphs.

TABLE I. Categories for Properties of Particles Which Determine Their Behavior in Fluid Environments

Properties dependent upon the physical nature of the particles

Size, shape, density, crystal structure

Surface area

Size distribution of powders

Porosity, pore size distribution

Particle packing, packing and tap density

Flow properties, angle of repose

Permeability of packed beds

Thermal conductivity, expansion

Properties dependent upon the chemical nature of the particles

Electrical and magnetic

Stoichiometry, impurity content

Reactivity of solids

Surface energy, solid-fluid interfaces

Rheology of solid-fluid systems

Thermochemical

TABLE II. Physico-chemical Effects Important in Particle Technology

Physico-chemical Principles	Application
Rate of a diffusion-controlled reaction is proportional to surface area (Fick's law)	Cement hydration; sintering reactions
Small particles exhibit higher vapor pressure than larger ones (Kelvin's equation)	Sintering; ageing of crystalline precipitates
Adsorption of vapors by solids due to chemical or van der Waals forces	Surface hydration of MgO; surface oxidation of metals
Capillary condensation in pores or at points of contact between particles	Agglomeration of particles in humid atmosphere; cementation of crystals
Spreading and non-spreading related to surfaces of tension at three phase interfaces (Theory of Wetting)	Strength of compacts; metal-glass seals; wetting agents
Contact charging of dry powders of high surface area	Clogging during flow; friction; angle of repose
Electrical double layer theory	Flocculation and dispersion of fine particle suspensions; slips; cation exchange
Theories of rheological suspensions	Thixotropy and dilatancy; filtration; slip casting

PREPARATION OF PARTICLES

Because the production method may profoundly influence important properties of particles such as size, shape, and reactivity, the more common methods employed to produce fine powders will be discussed briefly. Common production methods include: (1) comminution, (2) precipitation, (3) atomization, (4) thermal decomposition, (5) vapor deposition and (6) thermal quenching. The first three of these will be discussed.

1) Comminution.- Comminution is the size reduction of solids through the application of mechanical forces. Powders that are produced by crushing and grinding are characterized by a size distribution, with the finest particles being but a few Angstroms in diameter. Several recent papers have been concerned with the dependence of size distributions on loading methods, fracture mechanisms, and flaw distributions. A number of size distributions have been proposed for comminuted products.

Theories of comminution are at present inadequate to account for the energy used in grinding processes. The efficiency of industrial grinding equipment is assessed by empirical relationships based mainly on performance of laboratory models. A number of size distribution equations are available to describe the products from grinding devices. The various "laws" of breakage, however, are presently considered to be less important than the application of computers to describe size distributions and rates of size reduction. Modern milling trends include the revival of interest in autogeneous grinding, improvements in mechanical and fluid energy mills embodying high speed and extreme turbulence, fresh studies of ball mill operation at super-critical speeds, and the improvement of the vibratory ball mill. Precautions must be taken to avoid contamination of powders during the comminution of ceramic raw materials.

Milling not only affects particle size and shape, depending upon particle cleavage and flaw density, but also will change the reactivity of the milled product in a subsequent sintering step. Just why the reactivity is affected is highly speculative.

2) Precipitation from solution.- The processes by which precipitation from solution occurs may be divided into two primary stages: nucleation, and the growth of the nuclei to macroscopic dimensions. Present theories of nucleation have their origin in the theory of spontaneous nucleation developed for crystals nucleating from a super-saturated vapor. Once a crystal has nucleated in saturated solution, its rate of growth depends upon several factors which include number and kind of imperfections, kinetics, and mass transfer at interfaces, and the presence of foreign impurities. By suitably controlling the important variables in crystallization, different particle size ranges may be obtained. To a large

extent, the shape of the resulting crystals is determined by kinetic factors. Shapes may also be modified by controlling the supersaturation or by adding hydrocarbons which preferentially adsorb to certain crystallographic faces.

3) Atomization.- The processes occurring during atomization are complicated. There is the problem of the determination and control of the original drop-size distribution. The quality of the product will depend on the residence time of the particles, their temperature and humidity history, frequency of collisions, and other factors. A given set of conditions produces a certain kind of powder. Much remains to be done in a study of these conditions.

PROPERTIES DEPENDENT UPON THE PHYSICAL NATURE OF PARTICLES

The physical as well as the chemical properties of an assembly of particles are important to the ultimate goal of any forming method, namely to achieve uniformity of properties and a reproducible microstructure. It is important to know which characteristics are of specific interest to forming in general and how these are measured. These have been listed in Table I and are discussed in the following paragraphs.

1) Particle size, shape, size distribution and surface area.- The influence of shape, size, and surface area of particulate materials on their behavior is not well understood. This is partly because the problems of the precise definition of size, shape, and surface area have yet to be satisfactorily resolved and partly because of the problems encountered in measuring these quantities. The state of knowledge relating to these subjects is far from satisfactory and much remains to be learned.

Particle size is usually defined by a single dimension. However, actual particles are irregular in shape so that any size designation becomes quite arbitrary. The average linear measure of the projection of many particles in a fixed direction is statistically a measure of the size range of the particles if the orientation is random and a large number of particles are involved. This is sometimes impractical so particle size is defined by the method used in its determination. Three common definitions of equivalent diameter are a) the diameter measured microscopically, b) the sieve aperture diameter measured with standard size sieves, and c) the sedimentation diameter measured by sedimentation methods. When the shape of the particles deviates greatly from a sphere, the three definitions do not correspond, and in fact may differ by as much as a factor of ten.

One of the most abortive searches yet made is for a means of defining the shape of a particle. To date, the most common definition of particle shape is probably that which is based on the equivalent diameter, the surface area, and the volume. If d is the equivalent diameter, the surface area and volume of the particle are then fd^2 and kd^3 respectively. The ratio of f/k is then a measure of the shape of the particle.

Particle shape is important not only in forming, but also in packing, mixing, rheology, flow from bins, sintering reactions, and the ultimate microstructure of products.

Of extreme importance to characterization of powders is the size distribution of particles comprising the assembly. Particles formed by chemical means are characterized by a fairly narrow range of sizes. However, comminuted products have a distribution of sizes ranging from the upper limit down to a few multiples of the unit crystal. Numerous size distribution equations have been used to describe the weight-size relationship for a system of particles. Some have theoretical bases; others are strictly empirical.

A minimum of two parameters is necessary to characterize size distributions. However, often an average diameter is used in an attempt to describe a system of particles of different sizes and shapes by one number. Average diameters are based on linear, surface, or volume measures, and should be used by taking into account the exact phenomenon of interest, i.e., average surface diameter is generally used in studies of surface phenomena and permeability of packed particles. Such average diameters should be used with caution since the same average diameter can be obtained from a variety of distributions.

Table III lists several of the common techniques used to determine the size of a system of particles. The ranges of particle sizes applicable to the method are also shown.

TABLE III. Methods of Sizing and Range of Applicability.

Method	Size Range, Microns
Optical microscope	0.25 - 250
Electron microscope	0.0005 - 5
Sieves	5 - upward (generally above 37)
Fluid sedimentation	0.5 - 300
Centrifugal sedimentation	0.0005 - 50
Light scattering and transmission	0.1 - 30
X-ray scattering	0.005 - 0.05

Measurement of the surface area to characterize a powder is of great importance to most succeeding processing. Adsorption, solubility, kinetics, and many other phenomena are functions of surface area. As a first approximation, it would appear that in connection with properties of powders, such as the density of packing or the strength of an aggregate, the geometrical surface, as obtained by microscopy, permeability, sieving, or any other form of direct measurement, is adequate. For systems which require a measure of internal surface, gas adsorption techniques must be used. Several new methods to measure surface area have been introduced, of which the BET method of nitrogen or krypton adsorption is widely discussed in the literature.

It should be pointed out that different methods of measuring surface area do not give the same results. Thus, the reported value of the surface area is dependent on the method used for the measurement.

2) Porosity and pore size distributions in particles.- Porosity and pore size distribution is of interest to the reactivity of a granule and, in particular, to the coherence of a formed piece. A number of problems regarding the porous nature of particles exist, such as how to measure the closed pore volume. Several ways to measure open pore volume are available; usually closed pore volume is obtained by a suitable difference method.

The simplest way to measure the distribution of large pores is by optical microscopy. The electron microscope has been used for smaller pores. The difficulty with these methods lies in sectioning techniques and the need to view large numbers of particles.

Pore volumes may be obtained, for example, by a mercury intrusion technique. By applying graduated pressures, mercury may be squeezed into a porous solid and from the change in mercury volume a pore size distribution is calculated. Other methods involve gas flow analyses and the determination of adsorption-desorption isotherms. Mercury displacement is of value for macropore distributions; the filling of pores with condensed gas is useful for pores in the 300 Angstrom and smaller range.

3) Particle packing, apparent density, tap density.- Particle packing may be defined as the science and technique of mixing together particles of sizes and shapes so voidage is kept to a minimum. The best measure of efficient packing is probably a measure of the void space of the compact. In many instances, void space is used synonymously with porosity of the compact although the porosity of the particles is not considered. The important bulk properties are a) apparent density, b) tap density, and c) compression ratio.

The apparent density of a powder is the mass of a unit bulk volume of the powder. Tap density is simply the apparent powder density after it is vibrated or shaken down by a standardized procedure. Compression ratio is given by the ratio of the height of the uncompressed powder to the height of the pressed compact again by a standardized procedure. These terms have been most useful to the control of pressure compaction methods.

Theoretical models using particles of known geometry have been useful for predicting the behavior or irregularly shaped particles when packed together. When a minimum void space is desired, graded size fractions of material are utilized. Only a limited amount of work has been carried out to ascertain the packing of subsieve powders.

4) Flow of dry particle systems. - The ease of initiating flow of a particle system or granular mass is indicated by its angle of repose. High angles are found for systems whose distribution has a top size of less than 100 microns; low angles are obtained for closely sized material over 100 microns in size. The angle of repose depends on the way in which the powder heap is formed. The dimensions of openings blocked by a powder increase with increasing particle size; cohesion of the granules will also cause blocking. The dimensions of blocked apertures vary in the same way as the width of the empty annulus which forms adjacent to the edge of an aperture through which the material flows.

The flow of particle systems is usually studied in terms of theoretical work on the mechanics of plastic systems and recent treatments suggest that a flowing powder is analogous to a medium having cohesive properties, frictional resistance, and granular structural resistance which may vary as deformation proceeds.

The flow properties of powders are dependent upon the transmission of forces through the system, particle size, shape, and distribution, resilience, cohesion, and state of the surface.

5) Permeability of packed beds. - The permeability of a packed powder is defined as the volume of a fluid of unit-viscosity passing through a unit cross-section of a packing under a unit-pressure gradient in unit-time. This is really a simplified statement of Darcy's equation and hence the unit of permeability is termed the darcy. The permeability constant is a function of many factors, such as particle size, shape, porosity, type of fluid, particle surface energy, contact angle, and surface area. In fact, permeametry involves the passage of fluid through packed beds to determine

surface area of the bed. The utilization of flow through packings to study particle packing, porosity, and suction potentials is rapidly gaining momentum.

6) Heat flow through packed beds or compacts.- Heat may flow through a packing, through the particles, or through the fluid which contacts the particles. If the particles themselves are porous the heat capacity of the granules varies and must be considered in relation to the bed as a whole. The study of the flow of heat through packings is important to various pressure forming methods such as hot pressing. Numerous mathematical treatments have been formulated, these originating either from classical and statistical mechanical approaches or from dimensional analysis.

PROPERTIES DEPENDENT UPON THE CHEMICAL NATURE OF PARTICLES

Only those chemical properties which are being quite widely investigated, such as the surface activity of powders, will be mentioned in this short summary.

1) Surface energy, solid-fluid interfaces.- When a homogeneous phase becomes discontinuous, such as at a solid-fluid contact line, then that interface which forms becomes the seat of an excess or deficit energy content. At the surface of discontinuity, surface atoms are not in their equilibrium positions because of a difference in coordination. The literature is filled with erroneous definitions of surface energy and surface free energy per unit of area. In many cases it is thought to be analogous to surface tension as measured for liquids. From a thermodynamic point of view, surface tension and surface free energy per unit area are equivalent for a one component system provided the adsorption of the solvent can be taken as zero. For a system of two or more components, the surface tension and the specific surface free energy are not the same. For example, in the relation between pressure drop across a curved surface and the surface tension, one must define the system carefully before the surface tension may be referred to as the specific surface free energy. Similar ideas hold when surface tension is defined in terms of other variables such as energy content. The concepts hold for solids, for liquids, and for gases.

It should be noted that the real surface stress of a solid is related to its surface tension, the difference being due to the position constraints on atoms in a solid. Also surface energy is some positive function of a surface area increase, so it follows that a powder of high surface area has a higher total surface energy. This may relate directly to reactivity and to solid-fluid behavior such as with a rheological suspension.

Calculations of the surface energy of solids have been made by a number of researchers but many uncertainties exist in these calculations. Experimental measurement of surface energies is difficult but many of the investigations now agree reasonably well.

When a solid is immersed in a fluid, a redistribution of species will occur until an electrical double layer is generated. The properties of the double layer, which can be thought of as an equivalent condenser, are still open to question, especially since the models used are based on the assumption that solids have no space charge layer. Considerable refinements of the theory have been made with electrodes which are almost perfectly polarizable. In the case of solids, the parameter that is of interest is the zero point of charge of the solid-liquid interface. The nature of the potential-determining species must be characterized.

Any movement of a fluid past a solid results in an asymmetrical distribution of charge in the fluid relative to the solid. Hence a potential gradient exists and a force acts on the solid in accordance with the sign of the surface charge and the direction of the field. The field acts tangentially to the plane of movement. Various phenomena are related to this principle, namely streaming potential, electrophoresis, sedimentation potentials, and electro-osmosis. Under certain conditions, the dynamic double layer which is present during motion of a fluid past a solid is related to the static double layer developed in theoretical models. In these cases, considerable information may be obtained.

The rheological properties of solids are significantly dependent upon the existence of an electrical condenser at a surface of discontinuity. The double layer is important to cohesion with a compacted powder.

2) Reactivity of solids; stoichiometry; impurities. - A reactive powder is one which has been given some physical or chemical pretreatment to improve its ability to react more favorably in some way. For example, a reactive powder will more readily densify during sintering as compared to the same kind of powder which has not been made reactive. It is a phenomenon connected with surfaces and has been referred to as surface activity or surface reactivity. The reactivity of a surface is altered by a change in surface area and surface roughness, by a change in the content of bulk impurities, kind of lattice defects, and number of defects, by surface distortion and surface composition relative to the lattice type, by the gaseous atmosphere in contact with the powder, by the degree to which the atmosphere dissolves in the solid, and by the composition of the reacting mixture of solids (presence of additives). Particle size and reactivity are closely connected.

Most ceramic powders are insulators or semi-conductors. Therefore, their electrical properties may be severely changed by impurities and by the relative proportions of lattice components present. Moreover, their solid state behavior will depend on these factors, which also pertain to surface reactivity. The self diffusion of a species or of an impurity will depend on whether the powder is an insulator or semi-conductor.

Generally, a powder is made reactive during its initial preparation, or is purposely treated in some fashion in the batching or forming stage to alter its reactivity.

3) Rheology of suspensions.- Rheology is the science of the time dependent deformation and flow of matter. Suspensions whose properties are dependent on the degree of flocculation or dispersion, i.e., the state of electrical double layers, exhibit different kinds of rheological behavior. This is very dependent on the properties of the solids, especially size, shape, and solid concentration, all of which affect the deformation/stress response.

Theoretical models of non-Newtonian suspensions have been developed for spheres in fluids and for other geometries. Considerable progress in theory of rheological behavior has been made in recent years.

4) Electrical and magnetic properties which characterize powders.- Electrical properties of powders will differ from those of a solid when they depend upon the transfer of charge, because conduction paths are broken up in the powder. The surface will be chemically different from the bulk, for there will be layers of chemisorbed atoms and impurities. Therefore, the surface resistivity will differ from that of the bulk particle. The resistance of the powder compact will be dependent upon such factors as relative humidity, temperature, contact resistance, and the number of contact points which depend in turn upon the surface topography. Moreover, dry powders are extremely susceptible to contact charging wherein a particle charges oppositely to that object with which it is in contact. The charging properties of the powders will depend upon whether they are metallic, non-conducting, or semi-conducting in character. Charging may occur by particle-particle contact or friction, by particle-container contact, or by both. The maximum charge obtainable is limited by the breakdown strength of the fluid phase in contact with the powder, unless a direct short to ground is present. The electric charges which participate in the transfer are electrons and ions. There is no direct proof that ions are actually transferred by contact charging.

The magnetic properties of powders are affected by the degree of packing, particle size, and specific surface area. Magnetic powders are usually considered to have a dipole electrical double layer at the surfaces. Recent techniques based on precession of these dipoles have been used to study adsorption on magnetic particles.

NEED FOR CAREFUL CHARACTERIZATION OF POWDERS

Preceding sections have attempted to define and summarize the state of the knowledge dealing with basic particle and powder properties and behavior. The Bibliography contains many references which are more specific and which deal with subject matter of importance to characterization but which have not been summarized here.

There are two classes of basic properties of importance to characterization, namely those associated with the particle and those which are a characteristic of the assembly of particles. For proper characterization of particulate systems, basic properties of particles and groups of particles must be classified and measured. It must be determined which of these are important to a given forming operation and to the behavior of the formed piece in a subsequent treatment. This kind of information will serve to assist in choosing the forming method which is best suited for the particulate system.

In a highly ideal case, a raw material would undergo a standard testing procedure to obtain data which would place it into a classification scheme. Depending upon how the material is classed, the forming operation would be chosen to produce the best possible and product with that material. The classification would include schemes to modify properties and behavior to fit the forming method where desirable.

B. COLD FORMING PROCESSES

by A. W. Allen

Cold forming processes are the traditional mainstay of the ceramic industry in which particulate materials are compacted to the requisite shape under dimensional control by the application of pressure. As has been pointed out in the preceding section, the forming process receives from the batch processing a system of particles organized into a complex agglomerate system. The response of this system to a specific forming deformation is determined by the compaction response of the agglomerate base material.

1. Compaction Response in Cold Forming

The common denominator to the many cold forming systems which have been utilized may be in delineating the deformation response of a particulate system to the application of the forming energy. The final result of this response should be the geometrically characterized shape. The total mechanism of the compaction process which starts with a particulate system and ends with a shaped piece might be termed the "compaction response."

In the process class called slip casting the medium for the transport of particles during the compaction process is liquid, usually aqueous. If the shape to be formed is complex in geometry, the fluidity of the system must be increased to allow maximum conformity to the mold or die cavity which dictates the shape. Such fluidity is produced by a combination of (1) increasing the liquid-solid ratio and (2) fluidizing the particulate system at low liquid contents with special additives. The particulate system responds as a liquid of controlled viscosity (usually non-Newtonian). Compaction in the system occurs when the liquid is removed at very low shear rates and at pressures which are essentially the static pressure of the liquid itself acting against a rigid (or semi-rigid), porous, permeable mold surface. A portion of the liquid is removed by the mold during this process but more remains to be removed in the early stages of heat treatment.

Aids to the slip casting process to remove liquid and control casting rates have taken the form of applying pressure to the slip, pressure casting, or vacuum to the mold, vacuum casting. Centrifugal casting may be placed in this same category where the centrifugal pressure from mold rotation forces the liquid out of the particulate system.

Note that a particular liquid-solid ratio is not specified. As will be seen later the detailed application of rheology has expanded the applicability of this method to a wide range of particle-liquid systems.

If the cross section is constant and it is desired to produce a shape in which the length to cross section ratio is large and variable, the application of pressure to force the particle-liquid system through a die orifice may be employed. This type of plastic forming, referred to as extrusion, requires that the particulate system respond to forming energy by plastic deformation. The application of stress deforms the mass beyond its elastic limit but without fracture and in a manner which allows retention of the formed shape after the forming energy has been removed. The liquid-solid ratio is generally lower than for slip casting but again rheological studies of the systems have produced plasticity by a combination of (1) control of liquid characteristics; (2) employment of special rheological additive aids; and (3) control of rate of application of forming energy (strain rate).

Compaction during extrusion occurs by plastic deformation in both the extrusion chamber and in the die orifice. It occurs rapidly at high shear rates and high forming pressures. Liquid removal must occur after the piece is formed, most likely during the first stage of heat treatment. An innovation here is the incorporation of a liquid removal step as an integral part of the extrusion process but immediately following the extrusion step. Heat-induced diffusion of the liquid to an evaporative surface is motivated by the use of infrared or radio frequency energy.

The plastic deformation response of such systems may also be such that a die cavity of variable cross section but fixed volume may be employed in an injection molding process. The compaction response here is one of plastic deformation but finally in compression as the particle system builds up back pressure on itself. Additives which act as rheological aids are the key to uniform transmission of forming energy to provide fill and dimensional control.

To avoid the subsequent problem of moisture removal and to enhance the transmission of forming pressure, thermoplastic or thermosetting organic resins have been employed in injection molding. The temperature-time dependency of plasticity in an organic liquid-particulate system thus allows control of compaction response. The temperature-time cycle of forming concludes with a stage wherein the liquid viscosity increases to rigidity either by cooling or polymerization (or combination of both). The type of resin selected may allow the entire cycle to occur near room temperature and be primarily time dependent. Resin removal now becomes a part of the heat treatment cycle, although for certain applications the resin may actually remain as the bond, the forming stage being the final step in processing the product. Plastic deformation is still the major mode of compaction response.

Coextrusion of two different particulate materials is possible if their compaction responses are matched carefully by rheological analysis. Correlating the flow rate with extrusion pressure has been used for this approach.

The extrusion process or filter pressing may be used to prepare the particulate system in a plastically deformable condition, the major compaction response occurring when a blank or "bat" is formed. The blank in this condition is subsequently brought to the required shape in an auxiliary forming step. Slight additional compaction occurs in this step, the response here being primarily to deform plastically to the intended shape.

Thus in jiggering one surface is formed by plastically deforming the particulate system against a mold or die surface with a profile tool which defines the second surface. Generally cylindrical or elliptical cross sections are made by automating the movement of mold and/or tool. If the mold is permeable a liquid removal stage may be part of the process, but jiggering is also done in rigid, non-permeable dies.

Plastic pressing also employs a plastically deformable blank previously prepared by extrusion. In this process the blank is compressed against a permeable die surface with a second permeable die surface. The compaction response is now one of plastic deformation in compression not unlike injection molding. Liquid removal occurs under pressure by enforced diffusion into the die material. Pressurized air blown through the die material releases the piece and expels excess moisture from the die (usually plaster). Jiggering and plastic pressing have been widely employed where automation is a requirement of production.

Plastic rolling is a more recent extension of the plastic pressing process. At low liquid contents this may be substantially powder rolling.

Pressure forming is a process class in which the liquid content is essentially eliminated or minimized to avoid the attendant problems associated with liquid removal. Compaction response is a more critical function of (1) packing density; (2) mode of application of forming energy; (3) die design; and (4) particle-particle interaction. Additives are most typically employed which fall in the group of lubricants to affect minimum particle-particle friction and die wall friction, as well as binders to strengthen particle-particle cohension after compaction. Compaction response is apparently one of particle packing and elastic deformation perhaps to the point of fracture at high pressure but low shear levels.

Where close dimensional tolerances are required and the liquid content is low, "springback" is sometimes observed after the forming pressure is removed. This may be related to recovery of elastic deformation which occurred during compaction.

Some distinction may be made between dry pressing at liquid levels between 0 and 5% and "semidry" pressing where up to 15% liquid may be employed. The liquid level is a materials dependent factor. Particulate systems in which clay minerals are prominent components would be a typical example of the latter. An inert particulate system like alpha aluminum oxide may not tolerate very large quantities of liquid. Again process additives may fall in a class of surface active agents which influence this solid-liquid interaction in the system and hence markedly influence compaction response.

In the pressure forming process class great concern is expressed for the uniformity of transmission of pressure in a particulate system which is designed to pack to maximum density.

This is the paradox of pressure forming which has led to a multiplicity of inventive techniques for die design and system operation to facilitate compaction response in a basically immobile material. Most of these are based on uniaxial or at best biaxial application of forming pressure directly applied through rigid punches, platens, etc. to the particulate system. The engineering in these operations is highly developed and well adapted to particular product classes.

Realization that the homogeneous transfer of energy to the material in the die was a basic limitation of pressure forming led to the development of the <u>isostatic pressure forming system</u>. Compaction response results from the fact that forming pressures are applied simultaneously to the continuous outside surface of the shape. The die is an elastically deformable material which approximately determines the external configuration. The interior shape is defined by a rigid non-deformable steel mandrel. Compaction response here may involve the entire stress range of elastic deformation through fracture and perhaps plastic deformation in the particles themselves because this process permits operation at higher pressures than other methods of pressure fabrication (10^5 psi. as compared to 10^4 psi.). This also has become a well engineered system used in high volume, high precision production operations. Recent attempts to automate the process have also been successful.

An earlier version which used a particulate system encased in a rubber bag and isostatically pressed in a hydraulic fluid (usually oil) pressurized in a conventional die cavity is often referred to as <u>wet bag isostatic</u> or <u>hydrostatic</u> pressing.

In all of these the superior compaction response results from the unique mode of transfer of energy to the particulate system.

If the liquid level is low enough (2% or less) conventional pressure forming in dies may be used to produce a preform which is subsequently isostatically pressed so the compaction response is accomplished with a low pressure step followed by a second pressure cycle.

Realization that compaction response is so greatly influenced by particle packing led to extensive studies of vibratory compaction. Compaction response is the result of reduction in particle to particle friction by control of (1) size distribution and shape of particles; (2) lubricating particle surfaces in a liquid medium which dampens violent interaction; (3) control of vibration frequency and power to fit the geometry and mass of the compaction die system. Here again vibratory techniques may be employed either as a preliminary step to another process or as the sole method of forming. The technique is particularly successful when the shape is fixed by preformed metallic cladding (e.g. vibratory compaction of fuel elements in zirconium alloy tubing or refractory brick in steel jackets). The compacted clad shape may subsequently be altered in geometry by another method of forming. Pressure forming may be employed for this purpose. Cold swaging refers to an area reduction operation on such clad ceramics in which not only the cross section is changed but additional compaction in the particulate system may result.

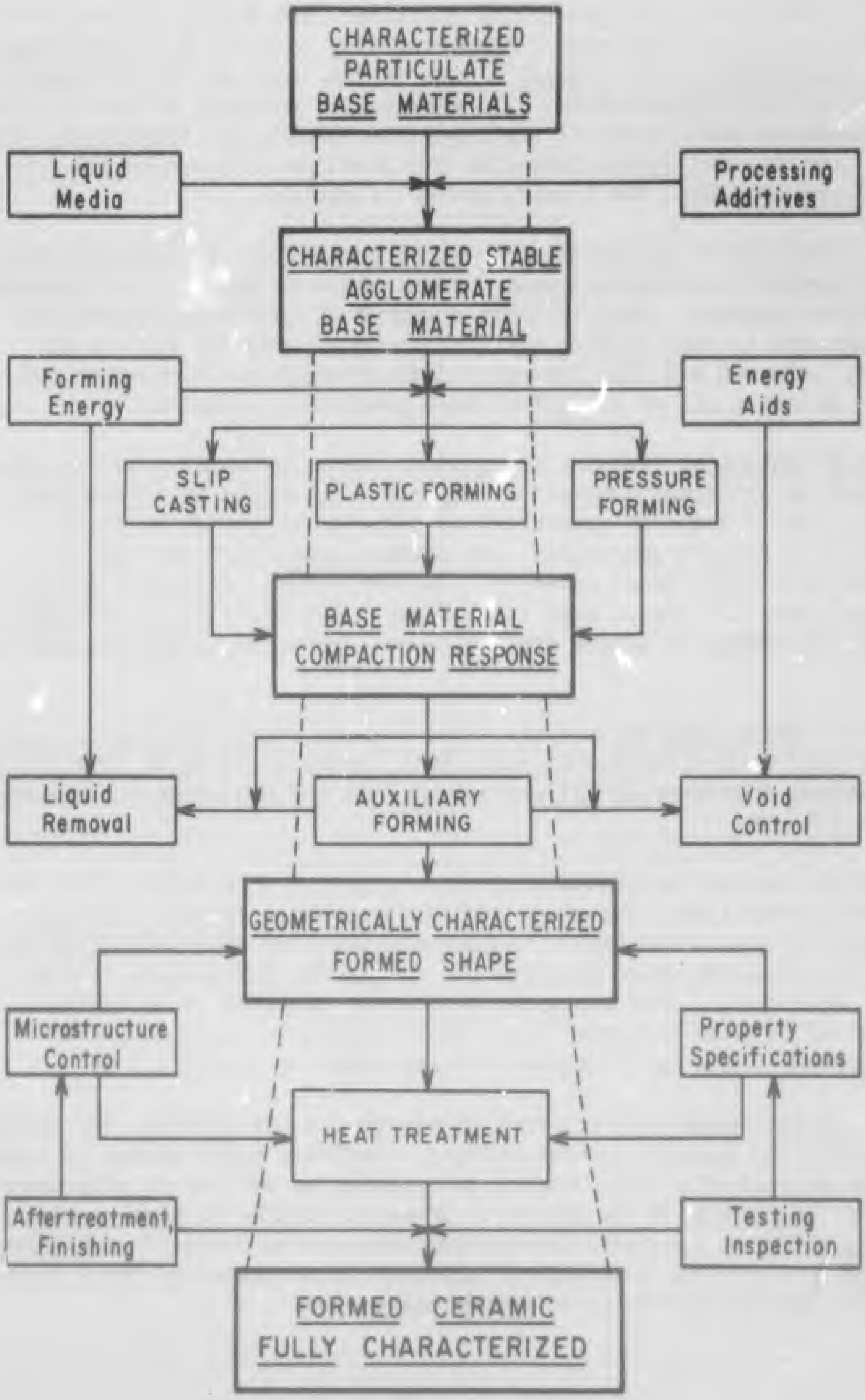
Many combination processes are possible in which one degree of compaction produced by, for example, extrusion is followed by a second operation which further compacts the particulate system and brings it to more precise dimensions. Repressing of extruded shapes in a pressure forming operation is typical.

Finally any of the processes may provide a shape which is brought to its formed dimension by any of a number of finishing processes such as grinding, sanding, turning on a lathe, etc. This may occur after the drying stage of heat treatment or in a stage prior to removal of all liquid added for forming, the so-called "leather hard stage." With the improvement of finishing abrasives, particularly diamonds, finishing of the final heat treated ceramic shape has also become routine practice.

2. Progression of Material Characterization in Cold Forming Processes

Figure 1 shows schematically the progression of characterization of materials in finally arriving at the end result: a fully characterized, formed ceramic. "Fully characterized" refers to the fact that ideally not only is the geometry known within specified tolerances but properties are known within specified limits of reliability. This requires, of course, a knowledge of the relation between the forming process steps and the heat treatment, finishing, and inspection steps. It should also be emphasized that in a few instances the forming step is the last stage in characterization when the requisite bonding and microstructure are achieved without heat treatment (e.g. "chemical bonding"), unless an auxiliary heat treatment occurs during the forming operation.

Starting at the top of figure 1 the first step is the characterization of the particulate base materials used, single phase or multiphase. Liquid



PROGRESSION OF MATERIAL CHARACTERIZATION IN COLD FORMING PROCESSES

Figure 1

media, often water, plus processing additives (see below) introduced in a proper mixing, blending, and/or tempering step produces a characterized stable agglomerate base material. This is the "mix" or "batch" which is handled in the forming system. The particulate material is now in a state of aggregation which gives it the requisite rheological properties, composition, liquid content, and particulate size distribution to optimize its compaction response when the forming energy is applied.

In addition to producing a degree of compaction, the forming energy may be expended to achieve liquid removal which is paramount to optimized compaction response. Here is where a number of inventive innovations have appeared in cold forming like centrifugal, pressure and vacuum casting; infrared and high frequency drying coupled to extrusion; and plastic pressing all of which have been previously mentioned.

Void control or removing the gaseous phase (primarily air) is also paramount to optimized compaction response. It should be emphasized, however, that a shape of characterized pore structure may be the objective of a forming operation. The optimum properties may not be achieved in a fully dense material. In other words "optimized compaction response" does not always mean fully dense. Forming a shape of high but known porosity may be a more difficult assignment than achieving maximum density.

Thus energy aids may include those forming system assistants which develop void control either to completely remove pores or to control them. (1) Chemical additives or (2) mechanical aids are two broad sub-classes in this category.

While chemical additives to produce pores or a stabilized foam can be readily envisioned, the class of additives which reduce entrapped voids may not be so familiar. Gaseous additives or solid additives of high vapor pressure which displace the air in the particulate system with a vapor phase which liquefies at forming pressure is an example. The mineralizers or sintering aids which facilitate reaction and/or grain growth in the heat treatment stages might be included here.

Mechanical aids take the form of either (1) air removal, (2) packing assists, or (3) control instrumentation. Deairing under vacuum is common practice in extrusion and pressure fabrication as well as in slip casting. Vibratory compaction as an assist to pressure forming is also widely practiced. Press equipment for dry pressing can be designed with almost any specified control over rate of application of pressure, dwell time and vibratory stroke during or before compaction.

Special mention should be made of ultrasonic aids because of their recent success on a pilot plant scale in ceramics. At relative easily attainable power level (50-500 watts) and frequency (60,000 cycles/sec.) a pressure insensitive transducer applied to extrusion dies for rods (¼" to ½" diameter) markedly reduced the power required and the liquid content needed for forming non-clay ceramics. A bell mode of vibration introduced at the die may act as an antifriction aid or may introduce forming energy at the point in the process where compaction response is occurring most rapidly. Ultrasonic energy applied through the mold to a thixotropic slip of very low liquid content has broadened the applicability of slip casting.

Some effort has been made to employ the feedback control loop approach to forming systems in general. In one instance the success of coextruding materials of quite different compaction responses has been the result of automatically monitoring extrusion rate as a function of extrusion pressure. If the extrusion pressure was controlled using the detection of flow rate as a basis, this control loop could be completed. In another instance detection of elastic strain in a vertical member of a toggle compaction press is used as a basis for automatically adjusting die depth and hence for controlling compaction response in pressing.

A secondary step in forming referred to in Figure 1 as auxiliary forming enhances compaction response and may be used to define the final geometry of the formed shape.

The particulate base materials have now progressed to a geometrically characterized shape. To this point the major problem has been producing geometric configuration with dimensional control. If heat treatment is included in the process the problem changes to one of maintaining dimensional control without geometric distortion. The traditional role of heat treatment is also to affect mineralogical and chemical changes which define phase placement in the microstructure. This may occur in prereacted materials which are discussed later.

The bottom of Figure 1 shows the links between the cold formed shape and the fully characterized ceramic shape to be microstructure and property specifications as modified by finishing treatments, testing and inspection. If the ceramic engineer could form to a characterized microstructure and if he had ultimate knowledge of the correlation of microstructure and properties this linkage would be precise.

3. Characterized Particulate Base Materials

Particulate raw materials with known physical and chemical (including mineralogical) properties are an obvious requisite to successful forming operation. A dilemma exists here because often the raw material supplier and the user are not sure where the responsibility for the characterization lies. This is true because the value of the product may not allow expensive characterizations of the complex system to the degree which is theoretically necessary. Empirical characterization and interdependence of supplier and user is thus a traditional situation. In the development of some materials (BeO and UO_2 for example) where costs were less critical, much fundamental information on characterizing the raw materials has been developed.

Of the properties of particulate systems considered important the following should be emphasized (see also Chapters II A and V E, the Bibliography):

(1) size distribution and shape
(2) surface area per unit weight and density
(3) crystal modifications, stoichiometry, defect structure
(4) chemical activity (e.g. hydration behavior, iodine number, anion impurities)
(5) degree of chemical purity or controlled impurity

Of these factors the size distribution seems to be the most universally important one to all methods of cold forming and to all product categories. In spite of the classical studies of particle packing the characterization of size distribution still appears to be at a semi-empirical stage because of (1) inability to <u>produce</u> stable particulate systems reproducibly in sizes below 10-15 microns; (2) complexity of the influence of shape factor in multiphase particulate systems; (3) poor reliability in measurement of particle size and shape, especially below 10 microns.

The abrasives industry is one of the few that has made an attempt to standardize specification of particle size and shape over the wide range of sizes necessary. This necessary range may extend from greater than ¼" diameter for some refractory products to less than ¼ micron for certain pure oxide products. Thus the engineer begins to reach a point of diminishing return in applying packing theory to the size formulation of particulate systems. This is complicated by the fact that in multiphase systems he may require several size distributions depending on the material component involved. The phase placement in the final microstructure may be related to this requirement. For example, in a recently developed process for improving high alumina refractory brick, secondary mullite formed at the grain boundaries of corundum was the result of incorporating "colloidal" silica in the particulate system. The familiar "gap sizing" used in the refractories industry is also an example of this increasing complexity.

So the engineer learns to relate the characterization of the particle distribution to a particular comminution process or mixing operation.

His characterization then becomes a function of the reproducibility of the process. The literature is replete with studies of particle size distribution (or more exactly equivalent spherical diameter distribution) as a function of dry milled versus wet milled versus energy milling or of the effect of calcining temperature on particle size, etc. etc.

Such a simulative, statistical approach may be his only economically feasible recourse. In fact, the most used tests for characterization of particulate systems seem to be of this simulative type. "Sintering behavior": press a pellet and see what happens when it is fired, or "compaction behavior": press a pellet in a standardized die and measure its compacted density. These are all realistic, expediant approaches to characterization. The extent to which characterization is fundamental may be a matter of degree of knowledge, and the criticality of characterization may be a function of the reactivity in the particulate system.

This kind of reasoning extends to the other particle properties listed above more than we like to believe.

One approach to minimize these variables is to prereact the particulate system, then reconstitute and carefully characterize the new material as a particulate system component. This prereaction may take any of a number of forms. The materials may be simply calcined to remove volatiles and dimensionally stabilize the aggregate. The entire formulation may be sintered at varied temperatures up to fusion to promote desired solid state or crystallization reactions and compaction. Or the materials may be completely melted to form a glass aggregate (called a "frit"). In certain instances the deposition or codeposition of phases by vaporization or precipitation from a liquid has produced a special characterization needed to improve beginning materials. In each case the resulting material could then form the sole particulate component of the forming batch or may be one of a multicomponent particulate system.

4. Characterized Stable Agglomerate Base Material

After the dry particulate system has been established it must be put in a stable rheological form acceptable to the forming process. The key to this operation is the addition of liquid and certain process additives which have a variety of functions. This is a highly proprietary area in which not only the kind of additive but the sequence of addition may be important. Criteria for selection become empirical and intuitive. Special granulating, agglomerating, or blending techniques also play a part here.

About all that can be done is to classify the additives as to function:

(1) binders for strength;
(2) lubricants for release, antifriction agents, or rheological aids;
(3) deflocculents and flocculents (soluble salts) for pH control or particle surface charge control;
(4) plasticizers for water retention, rheological aids, or strength during and after forming;
(5) wetting agents;
(6) water retention agents;
(7) antistatic agents for charge control;
(8) antifoaming or foam stabilizers;
(9) chelating or sequestering agents (ion retention);
(10) fungicides and bactericides for aging stability.

As an example, a good casting slip must (1) have controlled viscosity; (2) have good suspending capacity for solids; (3) have reproducible casting rate; (4) drain and release from mold; (5) have high strength after casting; (6) have known shrinkage when liquid is removed; (7) have complete stability with age. Obviously all these requirements will not be met with simple water-particle mixtures.

The ability to remove such additives in subsequent processing may control the selection of an additive and hence limit the entire forming operation. The thermal degradation of organic additives may be studied in detail as to (1) carbon residue; (2) ash content; (3) volume stability of formed shape during removal.

Reactivity between the liquid medium and the solid particles may present a problem in rheological stability which could carry over as a problem in heat treatment or as a problem in defining the chemical composition of the final material. For example, when the liquid medium is water and the solid material is a chemically active oxide like CaO or MgO, hydration readily occurs to change the chemical and physical constituency of the particulate system. Such an effect is exaggerated in the small particle fraction of the particulate system. The stability of casting slips of these oxides is very definitely affected by hydration. Reducing the temperature during all operations is one method of reducing activity. Employing liquids less reactive than water (e.g. alcohols) or additives which protect particles from hydration are other approaches to minimize the effect. Further complications arise in the removal of the water when hydrated oxides which are stable to temperatures in excess of the normal drying temperature introduce an added dehydration step in the heat treatment cycle. In certain composites where easily oxidizable metal particles are part of the particulate system, complications arise from oxide skins formed. Special mixing sequences or mixing in inert atmosphere may be necessary in these cases.

The complexity of this stage in preparation of the particulate system introduces the problem of characterizing the agglomerate batch material as input to the forming operation as a basis for control of the final product. Again an outline of the pertinent factors seems appropriate:

(1) Particulate size
    (a) mill product
    (b) granulated product

(2) Homogeneity
    (a) moisture and ignition loss
    (b) micro and macrostructure in formed shape
    (c) tracer technique

(3) Densification
    (a) bulk density
    (b) permeability

(4) Flowability and Formability
    (a) casting rate
    (b) granule flow rate
    (c) extrusion rate vs. pressure

(5) Green Strength-Density Correlations

(6) Sintering Behavior of a Standard Specimen

Any or a combination of these factors may be used to characterize the agglomerate base material in an attempt to predict how it will act during forming. A non-destructive, rapid test for green density and strength would facilitate this type of determination. (The characterization of particulate systems is further discussed in Section II A and references are cited in the Bibliography.)

5. Forming System Design: General Comments.

A thorough discussion of forming system design is beyond the scope of this writing because the recitation of historical developments would not be meaningful and because the significant contributions are either covered well in patent literature or are completely proprietary.

It is significant that factors such as (1) production rates, (2) production quantities, (3) continuity of demand, (4) safety considerations, (5) tooling amortization, and (6) required capitalization costs play a heavy role in the design of forming systems, perhaps an equal role with the requirements of the finished product or the characteristics of the material to be formed.

The problem of scaling up the production of a shape which is feasible in the laboratory is a particularly difficult one. A requirement for a forming system coupled to a heat treatment which would require a minimum of capital investment is a challenge given to design engineers particularly in producing large ceramic shapes.

The isostatic process development may be in this category because the capital investment per unit of forming energy available is substantially less than the conventional heavy press equipment. This is by virtue of improved efficiency of the process of transferring energy directly from a hydrostatic fluid or deformable die material to the particulate system.

Also the ultrasonic aids previously discussed are in the realm of improved efficiency in forming energy transfer.

One opinion holds that new concepts in forming system design will most always come from the intuitive, inventive person who may have broad experience but little formal scientific training. Support of development efforts in this category may be quite effective.

As the use of automated systems develops and the pressure applied in isostatic systems increases, a need for superior mold and die materials will develop. Automation places more stringent requirements on wear resistance and corrosion. Higher pressures in isostatic forming may be limited by the availability of suitable pressure vessels. Plaster as a material for molds may become inadequate as pressure casting, ultrasonic-assisted casting, and similar processes develop. Already the use of porous ceramic molds other than plaster, and porous metallic molds which are readily rejuvenated, are being explored. Low cost expendable molds may not be out of the question.

Another concept is that all forming system development should be oriented primarily to the end use. The development of radomes and of oxide nuclear full elements are two typical examples in which this is being done. This may be a much more realistic way of matching capital expenditures and development costs to the economics and strategies of the end product.

6. Geometric Characterization of Finished Ceramic Shapes.

Early in the survey it was thought that a limitation on size, tolerance or geometry could be developed for each cold forming process. Time did not permit a systematic survey. It became quite apparent, however, that such a survey would not have much value because theoretical limits are not recognized by the industry. Given time and funds most any fabrication job could be attempted with reasonable probability for success.

On the small end of the scale, barium titanate discs as small as 0.010" in diameter and 0.003" thick have been made by cold pressure forming. Hollow alumina porecelain cylinders 8 feet long and 27" in diameter have been made by pressure casting a slip. Graphite rod stock 105" in diameter is now available. 0.002" to 0.005" coatings have been applied to rod by co-extrusion and 0.003" holes have been held in rod only slightly larger than this in outside dimension. Almost any degree of finish or tolerance can be developed by grinding or polishing.

The smallness limitation seems to be somewhat more technically limited in that microstructure control looms as a barriar when the size of the part desired approaches the size of particles used or the grain size in the fired piece. Handling and transport systems need to be worked out for the small sizes. The economics of finishing (grinding) may dictate the degree to which very small parts can be brought to tolerance which is closer than ½% of any final dimension. All of these capabilities are well within the scope of the industry if the continuity of demand and/or price of the product allows it.

The largeness limitation is much more materials dependent. Large particulate sizes in certain oxides (e.g. BeO and $BaTiO_3$) have not been available, and producing an extremely large part from very finely divided material may be a definite limitation. The properties of a green formed piece may limit its size. If the green compressive strength is about 100 pounds per square inch how will it support its own weight if it weighs 2000 pounds? In the heat treatment process the ceramic usually looses strength in the early stages before it develops its coherent microstructure. All of these present handling problems which, while not insurmountable, may be expensive in their solution.

One very realistic largeness limitation is the availability of large heat treating furnaces. In the production of the 8 foot alumina cylinder mentioned a special kiln was engineered for this purpose. The problem of the large kiln is amplified when the temperature required is above 2900°F or the temperature uniformity demands gradients held to less than 10°F. Atmosphere control is an equally difficult problem in larger kilns.

So the largeness limitation is dictated primarily by the economics of translating precise technology to a large shape. It must be related to the total ceramic process rather than the forming process alone.

Better communication with designers who specify sizes, shapes, and tolerances may be a way of realistically modifying the requirements of geometric characterization. Often if the design is adapted to the material to a small degree rather then a complete attempt to adapt the material to the design, procurement is expedited without sacrificing any overall design requirements.

C. HOT FORMING PROCESSES

by R. M. Fulrath

Hot forming includes those processes for densifying ceramic powders or shaping a precompacted ceramic by the simultaneous application of pressure and heat. In a few isolated instances hot forming is used as a production method. The majority of the cases where hot forming is used, however, lie in the area of research and development. These methods of forming are unique in that they can be adapted to form many materials into high density specimens without extensive experimental studies. Because hot forming has been essentially a research tool, individual opinions on its utility and potential are often prejudiced by personal experience. This section has attempted to be objective in injecting the individual opinions of numerous workers in the field.

The evaluation of a forming method is primarily made on the basis of the properties of its product. The differences in the importance of a specific property to a technological area dictates the extent of interest in hot forming. For example, in producing oxide or carbide tool bits, high mechanical strength requires high density and a small grain size in the polycrystalline material. Hot forming by hot pressing has been developed to give these characteristics. In the development of nuclear fuels the density of compacted UO_2 grains is of importance. The utilization of this material does not require a high strength ceramic because of the metallic cladding used to form the fuel element. Hot forming by hot swaging or hot isostatic pressing can give a high density ceramic. The optimum optical properties of the alkali or alkaline earth halides require a high density. Hot pressing can again be applied to form such materials with the desired properties. The latter two examples do not require high strength in the compacted material, and, therefore, the hot forming methods applicable to these two technological areas may be broader than those applicable to the first example given.

1. Hot Forming Methods

Hot forming methods may be classified in a manner similar to those methods used in cold forming. In general, these methods are outgrowths of the cold forming methods with the added variable of heat. In a few cases the application of metal working methods has been tried to densify or shape ceramics and are included in this classification more to show awareness rather than give details of the application.

Because of the amount of information and experience available in uniaxial hot pressing, a major portion of this section will be devoted to this method.

TABLE I. Methods of Hot Forming Ceramics

(1) Hot pressing
    A. Uniaxial pressure application
    B. Biaxial pressure application
    C. Triaxial or isostatic pressure application
        (1) Pressure mechanically applied
        (2) Pressure applied through a gas or liquid medium
(2) Hot coining
(3) Hot rolling
(4) Hot extrusion
(5) Hot forging
(6) Hot swaging
(7) Methods involving high rates of pressure application

2. Uniaxial Hot Pressing

This forming technique has been applied to practically all of the oxides, nitrides, carbides, borides, beryllides, and halides. In the case of boron nitride, iron carbide, aluminum nitride, silicon nitride, and titanium diboride uniaxial hot pressing has been the forming method that produced compacted material with acceptable properties. A variation of the usual method of uniaxial hot pressing has been used to semicontinuously produce aluminum oxide rods.

The equipment and methods used in uniaxial hot pressing show considerable variation. Practically every individual working in the field has constructed his own hot pressing system. Each hot pressing system has its own characteristics with regard to its operation depending on the individual design. Recently engineered apparatus for uniaxial hot pressing has appeared on the market indicating the surge of interest in this technique.

A general description of a typical <u>uniaxial hot pressing system</u> is included and variations of this system described. In the simplest uniaxial hot pressing system the material to be hot pressed is placed in the center of a cylindrical graphite die. Cylindrical graphite plungers are inserted and the die assembly placed between platens of a press with suitable refractory insulators between the graphite plungers and the platens. Carbon black, carbon wool, or alternate insulation is packed around the die assembly and retained in place by a nonconducting shell of fused silica or cement bonded asbestos. An induction coil around the outer shell is used to heat the die assembly. Argon or other inert atmosphere is circulated over and through the thermal insulation to prevent oxidation. The pressure is applied to the material by a hydraulic jack acting on the graphite plungers. Normally the die and material to be pressed is heated to the desired temperature and then pressure applied. Maximum temperature and pressure attainable depend on the particular installation and design.

The major variables encountered in hot pressing equipment are the die material and design, method of heating, atmosphere control, and pressing aids employed.

Graphite is the most common <u>die material</u> used for uniaxial hot pressing systems. However, other die materials are being used on a limited scale. Table II lists the materials being used or considered for use and limits of temperature and pressure at forming temperature considered to be attainable at the present time.

TABLE II. Uniaxial Hot Pressing Die Materials

Material	Maximum Use Temperature (in °C)	Maximum Pressure (in psi)	Remarks
Graphite	2500	10,000	Inert atmosphere required
SiC	1500	2,500	Reactive with many materials difficult to machine
TaC	1700	8,000	Expensive, difficult to machine
WC and TiC	1400	10,000	
W	1500	3,500	Easily oxidized
Mo	1100	3,000	
Al_2O_3	1200	20,000	Difficult to machine, needs extreme care in loading, low thermal resistance
ZrO_2	1180	?	
BeO	?	?	
TiB_2	1200	15,000	Expensive, difficult to machine
Inconel X Hastelloy Stainless Steels	1100	varies	Mainly used for halides

Graphite is the most universally used material for hot pressing dies. It is available in many grades with differing densities, strengths, and thermal and electrical properties. Its increase in strength with temperature, low thermal expansion, ease of machining, and low cost make it a nearly ideal die material. Experience with many grades of graphite has not suggested any criteria for selecting one grade for hot pressing dies. Generally, the 1.6 to 1.8 gm/cc density extruded graphites are used. Molded graphites rather than extruded grades are considered best when the die is heated by electrical resistance. New materials such as the "ZT" grades of graphite and pyrolytic graphite are being appraised as possible hot pressing dies and plungers. Because of the enhanced anisotropic properties of these materials, their applicability to hot pressing dies appears to pose serious problems in die design. "Glassy Carbon" is another new material that may be investigated, though cost and availability of significantly large sizes presently limits its applicability.

The carbides have been tried as hot pressing die materials on a very limited scale. The expense of machining may be a limiting factor. The refractory metals tungsten and molybdenum have been used, but creep of the metals at high temperature limits their usefulness. Titanium diboride although showing excellent high temperature strength and an increase in strength with temperature (35,000 psi flexural strength at room temperature and 60,000 psi flexural strength at 1600°C) is limited because of the difficulty in machining, and, consequently, few experiments using TiB_2 as a die material have been attempted.

The alloys based on iron-nickel-chromium or nickel-chromium have been used extensively in hot pressing the alkali and alkaline earth halides. Their maximum temperature limitation is due to metallic creep.

Interest in oxide die materials for hot pressing in oxidizing atmospheres has increased significantly. High density alumina has shown promise in this area and can be operated up to 40 hot pressing cycles, provided extreme care is used in the operation of the equipment. Both alumina and stabilized zirconia are limited by low thermal shock resistance and creep at elevated temperatures; however, the lack of chemical reactivity with some materials and oxidation stability compensates for these limitations.

Design of hot press dies is generally based on the standard formulas for thick-walled cylinders with internal pressure. For small diameters these are generally successful in predicting maximum pressures attainable at any temperature, provided the tensile strength-temperature relationships are known for the die material. As the die internal diameter increases, imperfections in the die material make the behavior more erratic. This is especially true for large graphite dies where there has been considerable experience with hot pressing in the metallurgical field.

Die washes, liners, or sleeves have not been investigated sufficiently to predict their potential. Metallic foils of platinum, molybdenum, and tantalum have been used in graphite dies to prevent reaction between the material being pressed and carbon. Success has also been attained in using a mold wash such as boron nitride on graphite. Molybdenum dies have been chromium plated and used to 1100°C successfully with materials that react with molybdenum. Complex designs such as alumina sleeves in stainless steel have been attempted for low temperature hot pressing (under 1100°C).

In the hot pressing of dielectric ceramics of the barium titanate type, a zirconium oxide powder has been used between the graphite die and the dielectric. The zirconia powder transmits the pressure from the graphite plungers to the dielectric powder and prevents graphite reaction with it. It is not known whether the pressure acting on the barium titanate is uniaxial or hydrostatic. However, useful products have been obtained.

Induction heating of graphite dies is a simple and economical method. This technique, however, is more difficult to adapt to controlled rates of heating and cooling and to assured temperature uniformity in the specimen. Even so, induction heating is used in a majority of hot pressing systems because of the high temperatures attainable. *Resistance heating* of graphite dies by direct passage of electric current has interesting applications. For oxide dies and in some cases graphite die assemblies heating by radiation from resistance heating elements has allowed closer control of temperatures. Temperatures attainable by this method in general are under 1800°C. The type of heating employed in many instances is coupled to the type of material being hot pressed and specific hot press design.

Atmosphere control in hot pressing is often necessary. Graphite below 2200°C must be operated in an inert atmosphere or vacuum. Above 2200°C an inert atmosphere must be used because of the vapor pressure of the graphite. In many assemblies the graphite is heated and residual oxygen reacts with the carbon black thermal insulation or die material to form carbon monoxide as the gaseous atmosphere. The low rate of oxygen permeation through the carbon black prevents serious oxidation. Other systems flood and continuously flush with an inert gas such as argon. In either case the atmosphere at the sample position is unknown, but probably in the first stages of hot pressing is reducing.

Vacuum hot pressing has been attempted successfully in a number of laboratories. In pressing oxides one would suspect that an applied vacuum would facilitate attaining very high densities. There is no direct experimental evidence to confirm this. When pressing two-phase systems, such as glass-crystal combinations, there is definite experimental evidence that vacuum hot pressing reduces entrapped gas and leads to more uniform microstructures.

To aid the densification process during hot pressing controlled impurities are often added. Fluorides are added to oxide materials such as MgO and BeO. Post-forming heat treatment in oxidizing atmospheres of these materials can be used to eliminate the fluoride. This method has been used to produce translucent MgO.

In all hot forming processes the *response of the material* to the application of pressure and temperature is the most important parameter. Unfortunately, this response is a major unknown. Because density attained is the most universally accepted measure of hot pressing success, numerous materials have been investigated as to the density achieved under various pressure-time-temperature conditions. These relationships have been used to formulate the mechanism by which materials densify. Two major mechanisms have been proposed. One regards the mass transfer to occur by volume diffusion and the other by plastic flow. It is not apparent at present that either method alone is the only mechanism. It is possible and quite probable that particle sliding, plastic flow, and volume diffusion each take place during hot pressing and their relative importance in achieving a final density is governed by the character of the initial powder and the exact hot pressing environment. No worker in the field can or will predict the hot pressing conditions necessary for a given crystalline powder unless he has previously tried to hot press it. The general consensus is that a "try and see" attitude must be taken at the present time.

There are numerous examples of the same material chemically behaving quite differently according to the character of the crystalline powder. Fine particle sized (below 0.5 micron) alumina will compact to approximately 30 percent of the theoretical density under a load of 1000 psi, whereas, larger sized particles (above 2 microns) will pack to approximately 50 percent of the theoretical density. On heating these materials to 1000°C under 1000 psi applied load, the large particle size alumina will essentially retain the same volume, whereas, the smaller particle size material will densify to 50 percent of the theoretical density. No evidence of sintering or grain growth can be found in the fine powder. This difficulty in understanding the response of a material to hot forming in general has kept the field active in research and development. The ultimate ability to characterize fine powders and to determine the characterized powder's response to the simultaneous application of pressure and temperature is paramount to the development of a scientific approach to hot forming.

Experience has dictated the <u>primary process control in hot pressing</u> to be the pressure-temperature relationship. Most systems are operated so that the full pressure is applied at the maximum temperature. During the heating cycle a small holding pressure is applied to maintain alignment of the system. Other than this very little work has been attempted in evaluating the temperature-time relationships or effects of atmosphere control. Because uncompacted powders have low thermal conductivities, it is possible that severe thermal gradients would exist in the material to be hot pressed just prior to the application of pressure. In vacuum hot pressing time would also play an important role due to the slow permeation of gas through graphite or the powder to be hot pressed. These factors will increase in importance as the size of the hot pressed shape is increased.

Pressure distributions undoubtedly occur in hot pressing similar to those encountered in cold pressing. No information other than density variations in limited samples is available to support this conclusion.

It is realized that this would become increasingly important as sizes of hot pressed shapes are increased.

Hot pressed shapes, sizes, and uniformity are important considerations. Cylindrical, square, and rectangular cross sections have been fabricated. By use of a collapsible graphite tube to form the inner diameter, oxide tubes have been formed by hot pressing. As a "rule of thumb" the length-to-diameter ratio for hot pressed specimens should not be greater than 4 to 1. Although specimens with ratios as high as 9 to 1 have been formed, density variations along the length become increasingly severe above the 4 to 1 ratio. A unique method whereby the hot pressed material itself forms the bottom plunger has allowed alumina rods 4 inches in diameter and up to 6 feet long to be formed on a semicontinuous basis. The density of the product in this case varied between 90 and 95 percent of the theoretical density.

Examples of maximum sizes that have been hot pressed to this time are given in Table III.

TABLE III. Maximum Sizes of Hot Pressed Shapes

Material	Diameter in inches	Length in inches
BN	14	14
BeO	10	10
	12	2
Al_2O_3	9	6
MgO	7	1/2
TiB_2	6	18
	8	8

It is by no means implied that the sizes in Table III are the maximum possible sizes attainable. The general opinion is that if a larger shape were desired, it could be made by hot pressing if suitable equipment were available.

The smallest size that can be attained by hot pressing an individual shape is thought to be approximately 1/4 inch diameter by 1/4 inch high. No clear-cut limit is apparent, however.

Uniformity of hot pressed pieces either piece to piece or within one piece has not been subjected to thorough study. With beryllia density variations of ±0.2% in one piece and ±0.5% in repeated specimens have been reported. Alumina tool bits are quoted as having better uniformity from hot pressing than from cold pressing and sintering. Grain size variations in one beryllia sample 12 inches in diameter varied from 10 microns at the center to nearly 100 microns at the edge. The number of pieces repetitively produced by hot pressing to date has not been sufficient to adequately evaluate the uniformity of grain size, density, and other properties that can be achieved.

Various schemes to make hot pressing more economical by a continuous or volume process have been proposed. Die assemblies maintained hot with a heated feed and hot ejection have been suggested, but there is no evidence for such a systems having been tried. The use of many die assemblies which are preheated, passed under a press, and then cooled has been proposed but die costs are a prohibitive factor. Stacked pressing of a number of pieces in one die and gang pressing of a number of die assemblies have both been used successfully on a semicontinuous process.

The pressing of large shapes and cutting of the desired specimens from them is practiced by the producers of boron nitride and ferroelectric ceramics. This method has been limited to those materials for which cutting costs are not prohibitive.

Continual production by hot pressing does not appear to be impossible but superficial examination of the problem indicates unit costs would probably be high.

3. Other Hot Forming Methods

Little experience with biaxial pressure application at temperature is available, although the method has been tried in systems similar to conventional uniaxial pressing systems. No apparent gain was realized.

Interest in isostatic or triaxial hot pressing has significantly increased during the past five years. At least four laboratories are actively pursuing this forming method. The current interest is in using a gas for the transmission of pressure. In high pressure geophysical research the heated tetrahedral mechanical press has been used, but little information concerning its application to ceramics is available.

The equipment used in hot isostatic pressing consists of a pressure vessel containing a furnace. The material to be pressed is sealed into a metallic container, inserted into the furnace, and heated to the desired temperature. The pressure of an inert gas in the vessel is then raised to the desired level. One small isostatic press has a chamber 1-1/2 inches in diameter and 3 inches long. It can reach 2200°C with a pressure of 40,000 psi.

Large installations capable of forming samples 13 inches in diameter and 3 to 4 feet long have been constructed. These operate to 1550°C and 15,000 psi helium gas pressure. Canning materials to contain ceramic samples have been primarily of stainless steel.

Hot isostatic pressing has been applied to ceramics, cermets, and other composite materials containing ceramics. The method offers a unique pressure pattern which may be expected to cause material to respond to temperature and pressure differently than other hot forming methods.

Hot rolling of crystalline ceramics for the production of plates or sheets has been investigated in a number of laboratories. An extensive research effort is now in progress at one laboratory. The equipment that is being and has been used is essentially metal working rolls with alumina ceramic sleeves. Internally heated rolls have not been used; rather, the material is precompacted and heated then passed through rolls which are at a lower temperature than the compact. This results in undesirable heat transfer from the material to the roll. Evidence from barium titanate, forsterite, and synthetic mica indicates that a glass phase must be present to successfully hot roll ceramics. A porcelain body has been successfully rolled by preheating an isostatically preformed body to 1520°C and passing it between alumina ceramic rolls. A 25 percent reduction in thickness per pass was achieved. The glass viscosity and proportion appear to be important parameters in hot rolling. Twenty to 40 percent glass is necessary with approximately 30 percent optimum.

Hot Extrusion of canned and unrestrained ceramic powders has been investigated in many laboratories. Lithium fluoride powder has been hot extruded in vacuum to give translucent rods approximately 1/8 inch in diameter. Fine grain sizes were not attained. The extrusion of a single crystal of lithium fluoride gave a glass clear polycrystalline rod of fairly large grain size. The more refractory materials such as oxides have generally been restrained and coextruded with a metal can. Magnesium oxide has been hot extruded in a heavy molybdenum can. The oxide material exhibited a fine-grained surface and coarse-grained interior.

Marble dust canned in steel has been extruded and hot rolled between 700 and 800°C with no evidence of a liquid phase. Barium titanate has been canned in stainless steel and hot extruded at 1000°C with pressures of about 270,000 psi. A research effort on the hot extrusion of uranium dioxide both clad in stainless steel and unclad was recently initiated. The work to date has shown that at approximately 70% of the melting point in degrees Kelvin sufficient plasticity is achieved for hot extrusion. Density of the final product is the major criterion; however, a wide range of process variables will be studied.

Hot extrusion has posed serious problems on extrusion die materials. The high pressures required have usually necessitated that the material to be extruded be preheated and rapidly placed in a relatively cold extrusion chamber and extruded through relatively cold dies. What effect this has on the material response is unknown and may be a limiting factor.

The major problems with hot extrusion are associated with the lack of high temperature extrusion dies and the inability to characterize the material response.

Hot forging of cermet materials to both densify and shape has been successfully applied. Uranium dioxide-molybdenum and friction materials in which ceramics are dispersed in metallic matrices are two examples. Hot forging has been used to improve the density of fused silica. No evidence exists of hot forging being successfully applied to pure crystalline oxide ceramics. The response of either precompacted ceramics or powders to the relatively rapid application of pressure in forging appears to make this hot forming method impractical for this class of materials. The ductility imparted by a metallic or glassy matrix is necessary. The lack of availability of impact resistant platens and hammers for high temperature use would certainly make this area difficult to pursue.

Hot coining has been used extensively in the metal working field to impose a change in configuration on a preshaped blank. It has possibilities in ceramics for densification, changing shape, and embossing.

The application of hot swaging to unrestrained ceramics for densification is similar to hot forging and does not appear to have been tried. Metallic clad uranium dioxide has been hot swaged to densities above 95 percent of theoretical in forming nuclear fuel elements. However, less expensive and more reliable methods of achieving a high density have discouraged extensive investigation in this area. One investigation in hot swaging clad barium titanate resulted in a dense but cracked ceramic on stripping the metallic cladding. The compaction of electrical insulation by swaging is of course a well established technique.

Methods of hot forming involving high rates of pressure application have been studied. Cold forming of ceramics and cermets have been attempted by explosive methods. The experience to date has shown some promise when the material contains a ductile matrix. This method has not been tried on preheated ceramics or cermets. Pressure application in the millisecond instead of the microsecond range encountered in explosive forming has been achieved by use of gas accelerated plungers. This method has been tried to both densify and to extrude uranium dioxide and a few selected cermets for nuclear applications. UO_2 compacted by this method at 1100 C is reported to give large grains. No evidence of forming a structural ceramic by this method has been reported.

4. Summary of Hot Forming Methods

Hot forming ceramics by uniaxial or isostatic hot pressing is presently being quite actively pursued. The full potential of either method has not been exploited due to lack of studies in process control and the inability to characterize the response of materials to the simultaneous application of pressure and heat. The materials subjected to hot forming are often judged by one physical property because of specific technological interest. More detailed study of all properties are needed to adequately assess the forming method.

Improved die design is essential in both hot extrusion and uniaxial hot pressing. Production studies on equipment design by competent design engineers should assist in producing a continuous hot pressing scheme on an economical basis.

The kinetics of densification under simultaneous pressure and heat appears to be the important parameter in limiting the application of hot extrusion, hot rolling, hot swaging, hot forging, and hot coining to structural ceramics that do not contain a metallic or glassy phase.

D. MELT FORMING PROCESSES

by Alfred R. Cooper

Those processes in which at some stage, typically prior to the forming operation, the material is in a totally molten or nearly molten state are classified under melt forming. In Table I is shown an outline of the types of melt forming processes used in ceramics along with an indication of the types of materials and shapes that are produced. Each composition will exhibit its own characteristic behavior during processing. Because of the variety of processes, important material variables, and the rapid advances in the state of the art, it appears an overwhelmingly difficult task to hope to characterize definitively the limitations of all these processes for all materials and for all time. Instead the effort will be made in this section to suggest the types of limitations, the special features, the degree of understanding that seem desirable for fuller understanding of various melt forming processes.

Melt forming processes are divided into four major groups as indicated by the underlined headings in Table I. In general melting ceramic oxides prior to forming provides the opportunity to use a feed that is usually homogeneous and virtually without pores. It provides opportunity to obtain microstructures that are not achievable by any other processes. It typically allows fabrication to sizes and shapes that are only slightly modified by the shrinkage of the piece after forming. It presents an easy opportunity to achieve a wide degree of control of the atmosphere that is in contact with the material at high temperatures. Melting can often be used as a purification process. Finally non-equilibrium or high temperature phases with specially desirable properties can often be obtained only by crystallization from a melt.

Not only are there inherent advantages associated with melt forming, but clearly there exist serious disadvantages, some related to the fact that higher temperatures are required than in any other ceramic processing scheme, and others associated with the phase change from liquid to solid and the accompanying significant reduction in volume and the potential large change in gas solubility. Also, crystallization from a melt can result in segregations that negate the advantages of the original homogeneity.

TABLE I. Melt Forming Processes

1. <u>Glass Forming Processes</u>

   A. One Dimensional Objects
      (1) solid fiber
         a. melt drawn fiber
         b. melt blown fiber
         c. made initially as marbles, remelted, and drawn as fiber
      (2) solid rod
         a. gravity feed (Vello)
         b. extrusion feed (Danner)
         c. up draw process
         d. down draw process
         e. hand draw
      (3) hollow fiber
      (4) hollow rod (tubing)
         same processes as rod

   B. Two Dimensional Objects
      (1) sheet (three Process)
      (2) plate
         a. continuously rolled followed by grinding and polishing
         b. pot melted and poured
      (3) film

   C. Three Dimensional Objects
      (1) press
      (2) blow and blow
      (3) press and blow
      (4) casting (including centrifugal and injection)

   D. Coatings
      (1) flame spraying
      (2) room temperature coating with powder and melting (enameling)

2. <u>Crystallization of Articles Originally Formed as Glasses</u>

   A. Nucleated by radiation
   B. Nucleated by catalyst
   C. Self-nucleated

TABLE I. (continued)

3. <u>Forming from Melts of Non-Glassy Ceramics in Bulk (Fusion Casting)</u>

   A. Cooling in Place (in furnace)
      (1) to produce blocks for cutting to shape
      (2) to produce grain (refractory or abrasive)
      (3) to produce single crystals

   B. Pouring and Cooling in Molds
      (1) graphite compositions
      (2) refractory grain
      (3) steel
      (4) water cooled copper

   C. Continuous Casting

   D. Single Crystal Growing
      (1) vertical pull (Czochralski)
      (2) temperature gradient (Bridgman/Stockbarger)
      (3) floating zone (Pfann)
      (4) flux-grown

4. <u>Forming from Individually Melted Particles</u>

   A. Melt Sprayed Coatings
   B. Free Standing Shapes Formed by Melt-spraying
   C. Single Crystal Growing
      (1) flame fusion (Vernueil)
      (2) F fusion

1. Glass Forming Processes

Glass forming technology takes advantage of viscous deformations, a stable mode of shaping, involving a logarithmic increase in viscosity with decrease in temperature. Practical glass compositions permit transition over many orders of magnitude (10^3 to 10^{15} poise) of viscosity with decrease in temperatures to occur in a relatively short time interval during cooling to rigidity. The merits of the glass forming process lie in its wide flexibility to produce an extreme variety of shapes and sizes efficiently. Glass is virtually the most homogeneous of ceramics which among other advantages permits the polishing of surfaces to extreme smoothness (75 Angstrom units).

The ability to form a glass depends on the relative sluggishness of crystal nucleation and growth in super-cooled viscous liquids. It will depend therefore on the cooling rate achieved during processing. Thus, large articles will be more difficult to obtain as glasses and the compositions that can be utilized for glass forming will depend on the shapes and sizes of objects to be formed, and the method used for cooling. A rough indication of the viscosities of liquids that are suitable for glass forming under usual mass production conditions appears in Table II.

TABLE II. Minimum Viscosity at Liquidus Temperatures

spinning	2,000 poise
pressing	3,000
blowing	2,000
tube drawing	100,000
sheet drawing	100,000
sheet rolling	100,000

Those forming processes which cool the glass rapidly by having it contact metal mold surfaces with high surface heat transfer coefficients can tolerate much lower viscosities at the liquidus temperature than processes which inherently cool the glass more slowly because of the low surface heat transfer coefficients that occur at gas--glass interfaces. Not evident in Table II is the fact that surface area to volume ratio will also govern the cooling rate. Thus glass fibers can be drawn from relatively fluid liquids, while larger objects of the same composition will tend to crystallize.

Inherent Size Limitations. --The fundamental question as to how large a monolithic piece can be made from glass relates to this same heat transfer behavior. To produce a glass it is necessary to be able to cool the piece sufficiently rapidly to prevent the interior from remaining at crystallization temperatures for a period prolonged enough for devitrification to progress inward from its origin on the surface. As the rate of heat transfer of a slab is proportional to its thickness and the quantity of heat contained in the slab is also proportional to thickness, the rate of cooling of the interior is inversely proportional to the square of the thickness. Because of the inherent slowness of homogeneous nucleation, extremely low rates of cooling may be tolerable. In fact in certain glass systems there may be only little overlap of the temperature regions where crystal nucleation and crystal growth occur at finite rates. Depending on its nucleation and growth rates, each glass composition should have some limiting size to which it can be produced without devitrification. Little effort has been made to determine these limits but indications are that a thickness of 6" would be safely below the limit for most commercial glasses.

Limitations on Glass Utility. -- The utility of glasses is limited not by the forming processes but by (1) their behaving as viscously deforming liquids when heated to relatively low temperatures, and their (2) susceptibility to brittle fracture at lower temperatures. Since glasses invariably fail from tensional stresses at the surface, it is logical to attempt to improve their serviceability and strength by introducing internal stresses that will result in compression on the surface. This can be achieved in several ways as indicated in Table III.

The first process in Table III is most favorable for relatively thick cross sections of simple shape while the latter three processes can be applied even to thin sections of more complicated shapes.

Process 1 can be expected to increase the long-term strength of glass by a factor of four or more. Process 2 has reportedly achieved fifteen fold strength enhancements of standard abraded surfaces, but such techniques are mainly in the development stage and presently limited to a few compositions. Process 3 is equivalent to the application of a compressive glaze. Process 4, because its stress development does not depend on cooling, has possibly the widest potential. Unfortunately it is the least developed of the surface strengthening procedures.

Dimensions. --As mentioned earlier and indicated by Table I, there are a wide variety of ways of viscously deforming glasses to desired shapes and sizes at very rapid rates. In fact one of the fundamental advantages of glass as a material is the flexibility and speed of the glass-making process. There are however limitations in the size and shape that are attained by the equipment presently available as indicated by Table IV. While these limitations should not be considered fundamental they do represent what has been achieved and therefore represent an indication of the present limitations. As long as thirty years ago techniques for casting glass up to 200" were developed. The limitation was not the forming step but the annealing time, which required six months.

TABLE III. Methods for Inducing Compressive Stresses on Glass Surfaces

1. Cooling rapidly through the annealing range so as to have a "no stress" condition with a severe temperature differential between surface and center. When the glass cools completely to a uniform temperature, a continuous pattern of internal stresses develops from the unequal cooling with maximum compression on surface and maximum tension in center. This is called tempering.

2. Changing the structure or compositions of the surface region at elevated temperatures to have a lower coefficient of expansion than the bulk. On cooling compressive stresses will develop on the surface. Since the region treated is typically very thin, the compressive stresses are confined to an equivalently narrow region, and the interior tensions are of much lower absolute magnitude than the surface compression.

3. Cladding the bulk glass at elevated temperature with a glass of lower thermal expansion.

4. It has been proposed that diffusing large ions into the glass at the surface and thereby tending to expand the surface will result in the development of compressive stresses. Since this would be the only process that would not depend on subsequent cooling for stress development it would be the only process that would be of value for a low-expansion material like fused silica.

TABLE IV. Typical Dimensions for Articles Made by Glass Forming

Shape	Size
One dimensional solid	
*fiber	0.00012 ±0.00002" min. diameter
*extruded rod	2.0 ±0.10" max. diameter
cast rod	4.0" (or greater) diameter, limited length
One dimensional hollow	
*fiber	min. outside diameter unknown
*standard tubing	0.120 ±0.016" min. outside diameter
*standard tubing	7.0 ±0.150" max. outside diameter
hollow cylinder	14.0" (or greater) max. outside diameter
wall thickness	typical for 4" outside diameter tube 0.093 to 0.500"
**Two dimensional	
continuous ribbon	oil to 1.5" wide, 0.0014" min. thickness
continuous ribbon	up to 14" wide, 0.002 to 0.024" thickness
drawn sheet	up to 120" wide, 0.040" to 0.400" thickness
plate glass	up to 158" wide, 0.106 to 1.25" thickness
rolled glass	thickness up to 3"
Three dimensional	
small three dimensional articles fashioned from tubing	
pressed ware	30" diameter, 18" height approximate maximum; wide variety of single tapered shapes and sizes
blown ware	no shape limitation. 18" outside diameter; 30" length maxima
spun ware	limited to axially symmetrical shapes. 14" outside diameter; 36" length maxima

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*continuously drawn products; lengths depend only on convenience of cutting and/or winding

**all two dimensional products except rolled glass are made continuously so thickness depends on convenience of cutting. Thickness variations of 10% are typical.

2. Crystallization of Articles Originally Made as Glasses

Although the idea of utilizing the ease of the glass making process to form shapes and later on transforming them to different structures by heat treatment is relatively old, it is only very recently that this principle has been exploited in modern technology. Probably the first major example is the liquid-liquid separation that can be obtained by heat treatment of certain borosilicate glasses leading to two continuous phases. One of the phases being soluble, it can be eliminated by leaching. The remaining porous structure is readily sintered to form a non-porous glass. This technique has technological importance in that it allows relatively high temperature glass (96% silica) to be formed using ordinary glass making procedures.

Subsequent to this development there have been devised processes in which articles originally formed by glass-making have been crystallized either completely or locally and a new class of polycrystalline ceramic materials that have unique structures and properties have resulted.

An advantage of this process is that it utilizes the flexibility and speed of ordinary glass making to form the shapes. It therefore possesses the inherent merits of forming by viscous deformation. In addition the types of crystal structure and microstructure that can be achieved in crystallized glass go beyond those that are obtained by conventional ceramic processes. This is because the immobility of very viscous liquids allows nucleation and crystal growth to be separate processes. By controlling the temperatures and the sites for nucleation it is possible to nucleate different crystals. By controlling the time of nucleation it is possible to have the desired density of nuclei and thus determine the eventual crystal size. By controlling the time and temperature of crystal growth it is possible to obtain the degree of crystallization desired. This separation of nucleation and growth allows a wide variety of microstructures to be obtained. Particularly important is the opportunity to develop very fine structures with grain size as small as 200 Angstrom units. This results in transparent polycrystalline ceramics.

The homogeneity of the original glass material results in the development of polycrystalline structures that are extremely uniform from one local region to another. The process is controlled well enough to obtain reproducible microstructures. Typically, crystallized glass structures have zero porosity because the uniform crystallization and the remaining glass allow the structure to shrink during crystallization to compensate for any changes in volume that may occur on solidification.

Certain crystals that can be developed by crystallization from glass are difficult if not impossible to obtain by other ceramic processes. Some allow extremely low thermal expansion ceramics to be achieved. The fine microstructure is important for low expansion bodies because it tolerates the anisotropic thermal expansion typical of low expansion crystals. These low expansion bodies are extremely resistant to thermal shock. Finally, the small uniform grain size allows the development of polycrystalline structures whose strengths are relatively insensitive to surface damage, giving glass-ceramics significantly higher engineering strength than the glasses from which they were formed.

The presently available glass-ceramics are limited by the temperatures to which they can be utilized. The present maximum melting temperature is of the order of 1250°C and the recommended long-term use temperature has a maximum of 730°C. At the present the available compositions have involved the use of nucleating agents to develop the structures, and therefore the resulting composition has certain impurities from these nucleating agents.

In terms of their microstructures glass-ceramics have fairly wide flexibility. Single phase compositions have been produced, and compositions with as many as three or four different phases have been obtained. The amount of glass remaining in the structure can be varied over wide limits. Typically there is only one continuous phase. Because of the homogeneous nature of the original glass and the randomness of nucleation and growth, the crystals are entirely unoriented in the body. Grain sizes can be varied from as low as 200 Angstrom units to 10 - 20 micron, but generally the sizes are of the order of 1 micron. Porosities are usually vanishingly small.

The sizes and shapes that can be obtained in glass-ceramics are very nearly the same as those obtained by ordinary glass making procedures, Table IV. However, it must be recognized that the typical glass-ceramic compositions have greater tendency to crystallize than usual commercial glass and therefore if one wishes to control completely the crystallization process he cannot tolerate crystal nucleation or growth during the original forming process. This restriction undoubtedly limits the maximum size of the smaller dimension of certain glass ceramic compositions.

It is not known whether glass-ceramics have been produced from all shapes that are made as glass. For example, no indication of glass-ceramics in the form of fine fibers is presently available.

It is possible to utilize the principles of glass-ceramics in typical enamel coating procedures. Metals can be coated with glass by traditional methods and subsequently the coating can be converted by heat treatment into a polycrystalline ceramic that may have more desirable properties than the original glass. This procedure has also been used in the development of graded glass to metal seals where the thermal expansion is continuously varied by adjusting the degree of crystallization of a glass ceramic. Protective ceramic coatings on molybdenum metal that are said to be impervious up to temperatures of 1300°C have been achieved in a similar way.

Some of the processes described in Table III for inducing compression on the surface of glass can be utilized in glass-ceramics. Increases in strength from 20,000 psi to 35,000 psi, have been achieved in this way.

Localized crystallization of glass has been used as a process for chemically machining glass. In this procedure glass-ceramics containing traces of silver silicate are irradiated in local regions so as to favor nucleation of colloidal silver in that area. The resulting silver particles serve as nuclei for crystal growth when the material is heat treated at the growth temperature. If proper selection of crystal phase is made, the crystals can be dissolved or leached out by an acid that only slightly affects the surrounding glass. After the holes or other depressions are produced in this way, the remaining glass is totally radiated, nucleating colloidal silver throughout. A final crystal growth heat treatment converts it into a glass-ceramic. By this procedure holes of as low as 3 mils diameter can be obtained with a tolerance of ±1/2 mil. The geometry of the holes that are produced in this way can be either cylindrical or conical and perhaps even hemispherical.

While it has been reported that thousands of compositions have been demonstrated to produce glass-ceramics, there are only relatively few currently available. Extensive research promises to broaden the range of compositions and unique properties available by this process.

3. Melting and Solidification of Non-Glassy Ceramics in Bulk (Fusion Casting)

Fusion casting of ceramics currently has been confined to three areas, (1) production of relatively large refractory shapes for use in glass melting and steel making furnaces or for surfaces where unusually high abrasion resistance is required, (2) the production of small, high-purity, high-density shapes for nuclear reactor application, and (3) production of refractory, abrasive, and special purpose grain. While the nature of these processes is fundamentally the same, the size and the overall objectives differ drastically.

Fusion casting allows the achievement of localized regions of very high density (of the order less than 2% volume porosity) in extremely refractory materials. It is a process particularly suitable for the most refractory ceramics. The structure that develops on solidification frequently results in interlocking grains. These two characteristics produce a microstructure that is highly resistant to corrosion and erosion. It is possible to obtain extremely high purity in fusion cast materials, although often this possibility is not important. It is possible to utilize various atmospheres above the liquid during melting and in this way the stoichiometry and state of oxidation can be controlled. For extremely refractory, high-cost material it is possible to produce shapes of high strength and low porosity by fusion casting at lower cost than by the traditional ceramic processing. Uranium nitride and uranium carbide are examples of materials to which this rule applies. Extremely large shapes are routinely produced by fusion casting and the technology has developed so that shapes other than bricks, blocks or slabs can be produced. For example, large bowls and channels of various shapes are made for the furnaces of the glass industry by fusion casting.

An important advantage of fusion casting has been the opportunity to produce special crystalline ceramics such as beta alumina which is a sodium aluminate. This crystal is much more resistant to alkali atmospheres than is pure alumina itself. It would seem therefore that as a general principle it should be possible to produce by fusion casting ceramics that are specifically resistant to certain environments.

In spite of many advantages, fusion casting has inherent disadvantages which must be appraised with candor. Because of the brittle nature of ceramics and their characteristic thermal conductivity, there is minimum control over the grain structure and grain size that are attained from fusion cast bodies. In multiphase fusion cast bodies there is often a tendency for either direct or inverse segregation to occur. Thus both the composition and the microstructure of fusion cast bodies may vary significantly from the surface to the interior. The as cast surfaces produced by fusion casting usually have a waviness typical of the process. Casting void defects are also typical.

Methods of Fusion of Ceramics.--Melting of large quantities of ceramic materials and cooling in a controlled manner without pouring is an important process for the production of ceramic grains. Shapes may be cut from these ingots, but more likely they are carefully crushed to produce ceramic grain of controlled particle size and shape for subsequent processing. Much refractory and abrasive grain is produced in this way, as is the insulation for swaged electrical resistance heating elements.

If instead of cooling in place the melt is poured into molds, usually graphite or bonded refractory grain, the process is termed "fusion casting." While this is characteristically a periodic process, it has been found possible to continuously fusion cast both uranium compounds and ceramic oxides on a small scale.

Heating of ceramic materials on large scale for fusion casting has been done by immersing electrodes in the melt and more recently by the so-called "long arc" process in which the melt is used as one electrode and a relatively long arc is struck between it and graphite electrodes. This long-arc process allows for control of atmosphere that is impossible when the electrodes are immersed. It is also possible to use a disposable electrode that reacts with the atmosphere and in this way a very high purity melt can be obtained. Induction heating and electron beam heating are also potential methods.

The usual way of melting ceramic materials is in a shell of unmelted grain of the same composition which results in a non-homogeneous temperature distribution throughout the melt with the center being hotter and the edges being cooler. This temperature inhomogeneity to some degree is unavoidable. However in the long-arc process considerable stirring occurs which should minimize temperature variation.

It is reported that thousands of ceramic compositions have been successfully fusion cast. However, at the moment only relatively few are available commercially. This is probably because these compositions have been determined to have optimum properties for specific uses in furnaces for the process industries like steel and glass. Among the compositions presently available are magnesia, magnesia spinel, magnesia-chrome, zirconia-alumina-silica, alumina-silica, beta alumina, a mixture of alpha and beta alumina, chrome-alumina, uranium carbide, uranium nitride. There seems to be no reason why any oxide that melts at a relatively low vapor pressure could not be produced by fusion casting.

Typically the fusion cast refractory oxides have porosities of the order of 15 volume %. Most of this porosity is associated with a casting void or pipe that is ordinarily in the upper interior of the piece. At regions far from the casting void the local porosity is usually less than 2 volume %, and in the case of carbides it can be significantly less than 1%.

The pipe or void in fusion casting can be located with some degree of certainty by the positioning of the pour and by tilting of the mold, and in this way objects can be produced with the void in the least vulnerable area. If it is necessary to have fusion cast pieces without void it is possible to cast in a way that the void is all confined to a "riser". Then by cutting off the "riser" a sound casting is obtained. It is also possible to more or less distribute the shrinkage from solidification throughout the piece and thus avoid the presence of a large void.

The microstructure obtained in fusion cast materials depends on the chemical and physical properties of the material. Characteristically a reasonably fine grain size is obtained right at the surface of a fusion cast block where the melt has been rapidly quenched by contact with the mold. For some single phase compositions, immediately adjacent to the quenched surface there is found a region of columnar grains, some of which appear to extend almost to the center of the block or to the casting void. These grains are invariably oriented with their direction of rapid crystal growth in the direction of the center. In polyphase blocks the crystal size may be noticeably smaller as crystal growth of the primary phase can deplete the liquid of the components necessary for its further growth. Grain size in fusion cast ceramics will depend on the size of the block to be cast (increasing with increasing block dimensions) and the composition of the melt. They can be as large as a centimeter or two and sometimes as small as 30 microns. In certain compositions there is a uniformity of grain size from surface to center that may only vary by a factor of two. In other compositions and sizes a tenfold increase in grain size may occur from surface to center of the casting.

The more rapidly the block is cooled the greater the possibility of developing small size grains. However the rate of cooling is generally kept very slow in order to prevent thermal fracture during the cooling process. Hence it is usually only the very exterior which can be quenched. It must be concluded that in the present technology grain size is only moderately adjustable by varying the casting technique. In large shapes particularly, the grain size is mostly determined by the composition and the size of the casting.

Fusion cast oxide blocks typically contain from 0.5 to 17 volume % glassy phase. While this may be due to silica impurities or silica introduced to gain the proper compositions, the presence of a viscous liquid allows adjustments during solidification and during the early stages of cooling that diminish thermal stresses which tend to develop.

Data on dimensions, detailed descriptions of specifications, tolerances, squareness, and maximum sizes of various shapes are available from manufacturers. They are too specific and too extensive to list here. In the following only a few possibilities are mentioned.

Fusion cast blocks are currently cast in sizes as large as 18" x 30" x 48" with a weight of 3400 pounds from zirconia-alumina-silica and other compositions. In magnesia-chrome the maximum size presently made is 6" x 18" x 7". It is probably true that the presence of some glassy phase in the zirconia-alumina-silica composition allows it to be produced with a larger minimum dimension in reasonable cooling time. The surfaces of such blocks are typically rather rough and frequently hairline cracks from cooling are present. It is possible however to surface grind these shapes to relatively accurate dimensions and plane parallel surfaces.

The maximum size piece that can be obtained that is virtually free from porosity is about 8 cubic feet or essentially half the maximum size with voids. Plates 2" - 4" thick x 6" wide and 6' long are probably feasible to make in a virtually pore-free condition. Because of handling difficulties the minimum thickness typically made from fusion cast oxides is 1", but much smaller sizes are routinely cast with uranium carbide. Uranium carbide cylindrical bars of 1/2" to 3/4" diameter, 6" - 12" long are cast with a diameter tolerance of ±0.004".

Coating with molten ceramics has been proposed. Drain or slush casting has been achieved with molten ceramics in which steel cylinders have been clad on the inside surface by pouring in and then pouring out the molten ceramics. This suggests that it might be possible to obtain impermeable coatings for cladding of refractory metals by this process, or by dipping, or any other procedure in which molten ceramic was brought in contact with the cool metal surface.

Single crystal growing from liquid melts.--Unfortunately it was only recognized late in the study that such single crystals might be useful structural materials and no information regarding the melt processes for making single crystals has been obtained. Some references cited in the bibliography F11f will convey the present state of the art. Single crystal growing by melt spraying is discussed briefly in the last paragraph of this chapter.

4. Melting Particles Individually

Melt spraying of ceramics has been successfully utilized in two primary areas: (1) the production of high temperature coatings of high abrasion and corrosion resistance, (2) the production of free standing shapes, usually limited to shapes with axial symmetry. In the following there is presented a summary of the present state of the art followed by a discussion of some of the limitations. There is wide research and development interest in melt sprayed ceramics plus a developing industrial and commercial use. In the area of coatings alone one firm has three plants that are involved entirely with the application of coatings by melt spraying to a wide variety of shapes and materials. One list of commercial applications includes twenty-one items utilizing the features of wear resistance, thermal or electrical insulation, corrosion protection, or as a base for impregnation by resins.

In Table V is indicated the tolerances and maximum densities that are possible with flame and detonation spraying both for coatings and free standing bodies. In general it is seen that while coating thickness is limited there is no limitation to the size of objects that can be coated. In fact it is usually possible to coat by flame or plasma spraying in a discontinuous manner. While shapes of almost any configuration can be coated (corners and edges being especially troublesome), at present the free standing forms are limited primarily to such hollow axially symmetrical shapes as radomes, nozzles, crucibles, and tubes, or in general shapes with not more than one reentrant angle. If the substrate can be destroyed, greater flexibility can be achieved. The production of flat slabs is usually difficult because of warping. However zirconia discs for fuel cells of nearly perfect flatness were produced by melt spraying on sodium chloride and sintering the discs under weights to prevent warpage. Also 2" diameter alumina discs have been produced by spraying on an aluminum metal substrate, releasing by differential contraction, and sintering to cone 35 (1785°C). It is reported that discs sprayed to 0.025" thickness, sintered without distortion and were diamond ground to 0.015" thick, flat and parallel both sides to ±0.0005".

It is possible by flame spraying to coat wire of as small as 0.004" diameter and practical and convenient to coat wires of 0.030" diameter.

There is no question that melt sprayed coatings of parts that are especially subject to wear or abrasion can greatly increase the lifetime of the part. Objects as diverse as golf club irons, carving knives, plungers for glass pressing and wear surfaces on jet engine blades are sprayed for this purpose.

For axially symmetrical free standing shapes melt spraying has important advantages and convenience. Some of the relatively thin walled shapes easily attained with melt spraying cannot be produced by other ceramic processes. Large size free standing shapes are also reportedly easy to produce. Shrinkages on subsequent firing are lower than in traditional ceramics and the control of inside dimensions is most easily attained in melt spraying.

For very short runs or for the preparation of polycrystalline samples for experiments with refractory materials melt spraying seems to have special merit.

A wide variety of oxides have been successfully flame sprayed, including such materials as alumina, zirconia, chromia, zircon, zirconates, spinels, yttria, and ceria. The higher energy input and inert atmosphere capability of plasma permits spraying of an additional group of materials such as borides, carbides, and in fact all ceramics that do not have a high vapor pressure at their melting point. Some refractory materials such as beryllia, still present a serious problem of attaining satisfactory adherence. Empirical tabulations of the ease of flame spraying of various ceramics have been assembled. Such properties as heats of fusion, specific heat, thermal conductivity, and emissivity, are the material properties that influence the ease of flame spraying. Predictions of success with flame sprayed coatings cannot, however, be made by considering these properties alone.

In terms of purity, flame and plasma sprayed coatings are extremely useful, impurities introduced by spraying being assumed to be as low as 3 - 5 parts per million. In most instances it is possible to maintain stoichiometry during spraying but certain carbides require a sensitive control of atmosphere. This has been achieved with at least one coating technique. Also the high temperature oxides show some tendency to break down, but this effect is less serious.

In terms of structure melt sprayed coatings often contain but a single phase in addition to the pore phase. This phase may not be the equilibrium or most dense phase, for example in the case of alumina, the flame sprayed material is gamma or eta, rather than alpha alumina. In some instances, for example in flame spraying of zircon, there is a natural breakdown into two solid phases, zirconia and a zirconia rich silicate glass. Usually there is not an opportunity for reaction between powders in melt spraying. Thus in the melt spraying of mixtures of alumina and chromia powders there is no evidence of solid solution in melt sprayed deposits, although a complete range of solid solubility exists. If necessary one can produce more complicated phase distributions, for example, mixing of nickel and alumina powders or clading alumina powders with nickel metal permits the production of flame sprayed nickel-alumina cermets. A similar procedure is the flame spray of mixtures of pyrex glass and zirconia to give a two-phase system.

Flame sprayed materials have grains that are usually flattened in the direction of flame spraying. The degree of flattening depends on the velocity and the superheating of the sprayed droplets, detonation spraying perhaps giving the greatest degree of "squash." The grain size is determined in powder spraying almost entirely by the grain size of the incoming powder. Powders that are less than 5 microns are difficult to feed to flame spray or plasma spray guns. This factor limits the minimum grain size that is easily obtainable. Submicron powders have been fed into the detonation gun by utilization of special powder dispensing techniques. Also 0.1 to 0.01 micron spheres have been produced by decomposing organometallic liquids in the oxy-hydrogen flame, but this appears to have been a vapor process.

A serious limitation of the applicability of flame sprayed coatings is the presence of continuous pores in the as-sprayed state. Typical porosities of flame sprayed ceramics run from 3 to 15% with the difference depending on operating variables and upon the particle size of the feed powder. While most of the pores seem to be continuous or open, there is some evidence of a minor amount of closed pores existing in flame sprayed deposits. Attempts to attain impermeable coatings have been successful with lower melting materials like barium titanate, and special compositions that are impermeable are said to have been developed for coating molybdenum.

The factors that favor the development of impermeable coatings seem to be: (1) low melting temperature powder, (2) high substrate temperature, (3) uniform particle size, (4) fine particle size, and (5) uniform heating of every particle. Even when impermeable coatings are obtained their utility is still questionable. The brittle character of ceramics makes them susceptible to fracture from impact or heat shock. Microcracks can spoil the impermeability. Also when complex shapes need to be coated the corners and edges seriously compound the difficulty of attaining truly impermeable coatings by melt spraying. As-sprayed permeability is not as serious a handicap to free standing shapes because they can be sintered to an impermeable condition after spraying.

While it is usually impossible to heat melt sprayed coatings hot enough to sinter the coating to eliminate gas permeability, a nearly equivalent procedure is to subsequently impregnate the coating with glass after spraying. A recent development allows the glass to be sprayed concurrently with the ceramic particles. While this does not result directly in an impervious coating, sintering of the glass when the material is heated to operating temperatures causes the coating to become impervious by the time the surface protection is required.

When the feed material is introduced in the form of a sintered rod rather than as individual powders, the particle size distribution is determined by the turbulence in the plasma or flame. To date a technique of obtaining finer particle size than 10-15 microns from rod spraying has not been achieved. Rod spraying is said to have an advantage of allowing the residence time for each droplet to be greater and perhaps more nearly uniform than can be achieved in powder spraying.

Melt sprayed ceramics have been applied to a variety of materials including metals (with melting points equal to or greater than aluminum), other ceramics, graphite, resins and such soluble materials as alkali chlorides. It has been shown that the primary bond to substrate in melt sprayed ceramics is largely mechanical. Typically it is adherence that limits the utility of the coating, therefore it is desirable to roughen or even undercut the substrate to gain good adherence. Values for adherence of alumina and zirconia coatings to roughened substrates in tensile testing have ranged from 400 to 1300 psi. An interesting technique for achieving adherence is to melt spray a substrate with its own composition at low velocity and low superheat to form well bonded but only slightly "squashed" droplets on the surface. This reportedly provides a surface equivalent to undercutting which gives higher measured values of adherence.

For free standing shapes it is desirable to spray at temperatures as low as possible to minimize the possibility of adherence. Then it is necessary to use a high expansion metal that will shrink away from the coating. When it is necessary to spray free standing shapes at high temperature to achieve greater densities or strengths, there is a problem of finding suitable parting agents.

To date spraying has been done with either flame or plasma or acetylene/oxygen detonation as the source of the energy. Table VI shows some of the advantages and disadvantages of the alternate techniques. (Electron beam and radic frequency heating can also be utilized, but these are in the early stages of development and their specific advantages remain to be proved.) The feed is introduced as either rod or powder and comparisons are indicated in Table VII.

Deposit efficiency for flame spraying (deposit weight/feed weight) varies between 15 and 95 %, depending primarily on the fraction of the spray that is intercepted by the work and also upon the velocity and degree of melting of the droplets.

Flame spraying is a convenient way of making spherical particles. The minimum size is about 1 micron and the maximum size seems relatively unlimited, for example 500 microns. A difficulty with large spheres produced by flame spraying is that cooling from the outside results in an interior pore due to the volume change on solidification.

The manufacture of single crystals by spraying powders is a melt process. While such parts have been used for special optical and electronic purposes, structural uses also exist for single crystals. Compared with their polycrystalline counterparts, single crystals possess the advantage of higher thermal conductivity, freedom from gas diffusion, and less sensitivity to attack because of the absence of grain boundaries. Properly mounted crystals will not develop the thermal stresses that can be present, even at uniform temperature, in usual polycrystalline bodies of anisotropic materials.

Single crystals can be grown from all ceramics that (1) melt congruently, (2) have relatively low vapor pressure at melting point, and (3) have no crystallographic transformations between m.p. and room temperature. Single crystals can be grown under the tolerances and specifications shown in Table VIII. Ability to control crystal orientation at will has been a limitation.

TABLE V. Dimensions and Tolerances of Melt Sprayed Coatings and Shapes

Coatings	Flame Sprayed	Detonation Sprayed
thickness variation as sprayed	±0.002"	±0.0001"
maximum tapers as sprayed	±0.002"	
maximum thickness of coatings	0.050 to 0.375"	0.020"
maximum thickness of continuous coatings	0.002 to 0.004"	0.0005"
open porosity	3--15%	1%
finished thickness variation	std. grinding limits: ±0.0001"	+6x10-6" (special gauge block)
surface roughness as sprayed	200 microinches	150 microinches
surface roughness, finished	10 microinches	1 microinch
Free Standing Shapes		
thickness of axially symmetrical bodies	unlimited	not determined
length or width, normal to spray direction	unlimited	unlimited
linear shrinkage during sintering	3-4%	
inside diameter variation	1" ±0.0015" 1-2" ±0.003" 2-3" ±0.005"	
outside diameter variation	2x inside diameter variation	
open porosity after sintering	practically zero	

Notes: 1. Relative values do not indicate superiority of one process over the other

2. Deep cavities where depth is greater than diameter are difficult to spray if inside diameter is less than 3½"

3. Example of a flame sprayed free standing shape is alumina of 12" diameter with ±0.020" on both inside and outside dimensions.

TABLE VI. Some Features of Different Types of Melt Spraying

Flame Spraying

Cheapest

Easiest

Portable

Quickly set up

Useful for free standing shapes and coatings

Possible to coat inside diameters

Particle velocity 150-500 ft./sec. depending upon design of apparatus

Arc Spraying

Highest Temperatures

Wider control of atmosphere for protections of grains and/or substrate

Used for coatings and free standing shapes

Any material with low enough vapor pressure can be plasma sprayed

Possible to coat inside diameters

Particle velocity approximately 1200 ft./sec.

Detonation Spraying

Oxy-acetylene explosions about 4-9 per second

High velocities give best adherence and density

Pulsed energy and particle impingment reportedly gives least heating of substrate

Mostly used for eutectic-liquid-carbide combinations, alumina and alumina-chromia mixtures

Only used for coating, extremely noisy, large stationary installations

Particle velocity approximately 2500 ft./sec.

TABLE VII. Some Features of Powder and Rod Spraying

Powder

- Doesn't require additional processing steps (but is often prepared to close specifications)
- Allows more control of grain size
- Gives finer grain sizes
- Slower build up usually
- Cheaper for certain materials
- Can be used with plasma, flame or detonation
- More materials available

Rod

- Larger particles
- Longer resident times in high temperature zone is claimed
- Better adherence (in comparison with similarly heated powder) is claimed
- Faster build up of thickness
- Not practical with detonation

NOTE: Feed rods can be obtained as solid rods, fluted solid rods, and hollow rods.

TABLE VIII. Present Single Crystal Dimensional Limitations (Alumina)

- rods:
  - maximum diameter 1 1/2" or larger
  - minimum diameter unknown
  - maximum length 18"*
- discs:
  - maximum diameter 6"
  - minimum thickness 1/2"
- domes:
  - maximum diameter 6"
- sheets:**
  - maximum width 1 1/2" or wider
  - maximum thickness uncertain
  - minimum thickness 1/4"
  - maximum length 18"*

*This limitation is easily overcome because rods are formed continuously in this direction

**Called by crystal growers "boule sheet"

E. MISCELLANEOUS FORMING METHODS

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In the following sections several less common, but highly useful and strategic forming techniques are discussed. Each is undergoing intensive research and development because it contributes certain structures or permits flexibility in processing which can be realized by no other approach. The potential for these techniques is wide, but their usefulness has been limited by economic considerations in many cases. One limitation has been the low-volume demand for the products whose characteristics are uniquely set by the forming method--foamed ceramics, for instance.

The processes considered can be further studied through the references cited in the Bibliography under the classifications indicated:

1. Forming from vapors--F25
2. Porous or foamed ceramics--F16
3. Forming from fibers--F26
4. Bulk placement and molding--F2h, F2j, F2k, F3g, and F5b
5. Impregnation and infiltration--H16
6. Reaction sintering--F5g
7. High energy rate forming--F8
8. Electrophoretic forming--F27 and F7
9. Machining and grinding--all of G and specifically G4, G17, G18, G19

1. Forming from Vapors

Unusual ceramics not obtainable by other forming techniques are being formed by chemical vapor deposition. Chemical vapor deposition is a technique which can be termed molecular forming; that is, the building up of coatings and massive deposits by deposition of molecular or atmoic particles. A solid product is formed when the reaction of a suitable vapor is made to occur on a heated surface. The reaction used is one in which all of the components of the system except the desired product and the substrate are volatile at the deposition temperature. The product is of theoretical density and, when the reaction takes place properly, is impervious.

Chemical vapor deposition is accomplished in equipment usually termed a reactor. In its essentials, the equipment must include a retort in which to volatilize the feed material, a carrier gas to take a vapor to the working surface, a hot substrate on which to deposit the material of interest, and an exhaust. The equipment can be laboratory size or large enough to form structures suitable for missiles and rockets.

The source materials from which the vapor deposited ceramics can be formed fall in three categories: inorganic halides, purely organic compounds such as methane (used commonly for the deposition of pyrolytic carbon), and the alkyl and carbonyl families of metal-organics. It is appropriate to point out that some of the carbonyls are toxic and should be used with extreme caution. All of the halides, which are common source materials for vapor deposition of ceramics, are sensitive to moisture and oxidation. The oxyhalides thus formed are stable and are difficult to volatilize. The metal-organic compounds oxidize readily even to the point of being explosive.

A principle use for chemical vapor deposition for forming ceramics is for the production of high density, impervious coatings. Considerable effort has gone into the coating of nuclear fuel particles. Coatings of pyrolytic carbon, niobium carbide, silicon carbide, alumina, beryllia, zirconia, chromia, and mixed oxides have been made.

Mixed feed vapors can be used to form intimate-mixture deposits. In systems which normally form solid solutions, solid solutions will form by vapor deposition also. In two phase systems, microcrystalline products can be formed and crystal sizes kept small if the deposition is carried out at a sufficiently low temperature. Carbides are formed by the deposition of a metal on a carbon substrate with proper control of the temperature and pressure so that only the metal carbide is stable. The rate of carbide formation is controlled in such cases by the rate of diffusion of carbon from the substrate through the carbide coating, providing very uniform coatings.

The rate at which ceramics can be built up by chemical vapor deposition will vary, of course, depending upon the material and the requirements of its structure. Build-up rates of about 20 mils per hour are possible. Since ceramics formed by this process must be formed on a heated substrate, the shape of the ceramic is determined by the substrate shape. Since the reacting gas surrounds the substrate, the surface on which the ceramic is deposited can be of any shape. Thus, the shape of the ceramic itself can vary as desired. In making coatings, the gas flow must reach all surfaces as evenly as possible to provide uniform coatings. The gas flow can be controlled by baffles or jet streams. If the ceramic desired is to be free standing, the material is deposited on molds or mandrels which are subsequently removed.

The making of thick sections of either a coating or a free standing ceramic poses problems. Progress is being made in the production of thicker pieces, but thicknesses ranging from 1/4 inch to 2 inches, depending upon the material, still present considerable deposition difficulties.

2. Foamed Ceramics

An accepted method of producing very light weight ceramics is to foam the structure, and a number of foaming techniques have been used. The techniques include chemical reaction-hydrolytic reaction, reaction of acid or alkali with a metal additive, incorporating air by agitation, and oxide reduction and gas release. Not strictly a foaming process, but a process used to prepare a similar porous product is one which involves burning out organic additives. Other techniques of producing a light weight material involve bloating the material being fired and sintering of the preformed bubbles or spheres.

All of the foaming techniques are characterized by, at some point in the forming process, the formation of a thin-walled porous structure which is self-supporting and which is given its shape because it retains a generated gas. In such processes the rate of gas evolution must be closely controlled and must occur during a period of the process in which the structure can be stiffened to retain the outline of the foam after the gas generation is arrested.

A detailed discussion of one of the common foaming processes will illustrate some of the factors important in foaming. This foaming technique involves the use of resins. Three types of resins are used together--a phenol formaldehyde, an epoxide, and an isocyanate. The isocyanate resin provides the foaming action while the other two provide the necessary stiffness to the porous mass. The foaming action results from water in the phenol resin reacting with the isocyanate resin to produce carbon dioxide and some nitrogen. The reaction increases the temperature of the mass, causing the phenol and epoxide resins to polymerize and set to a firm structure, trapping the gas bubbles. Ceramic particles mixed with the resins prior to the foaming action are dispersed by the foaming and are left on the walls of the pores.

In controlling the characteristics of the foam, temperature is very important, as is the proportion of the resins. By varying the resin proportion, the strength, density, pore size, and bubble content can be varied and controlled. Control of the environmental pressure during the foaming also can be used to control the foam characteristics.

After the foamed article has been formed, the resins are carbonized to eliminate the volatiles and then the porous mass is given a final heat treatment to sinter the structure. If carbides are to be formed the carbon residue of the resins can be reacted with an oxide in the final heat treatment to sinter the structure. If carbides are to be formed the carbon residue of the resins can be reacted with an oxide in the final sintering treatment in an inert atmosphere. If carbon is not wanted in the final structure, it can be burned out during sintering.

In forming of foamed ceramics in which the foaming takes place at an elevated temperature, the temperature must be controlled closely to generate the desired pore characteristic. For enhanced control of the type of pore generation which takes place during firing, carbon or graphite particles have been added to the ceramic mix. Properly dispersed, they provide a source of gas in dispersed locations throughout the mass, leading to more uniform pore structure.

Foamed ceramics usually are of a simple geometry as fired. The fired mass is readily machinable and can be shaped as desired. The size limitation depends upon the foaming technique used but is less severe than in most ceramic forming techniques. Massive blocks have been made.

3. Forming from Fibers

Structures utilizing fibers are made in such a way as to take advantage of the fiber tensile strength. Typically, fibers having a high-elastic modulus are incorporated into a material of low-elastic modulus so that the fibers will absorb most of the tensile stress to which the structure is subjected. Also, there has been some use of fibers alone, so wound as to be self-supporting. By far the greater use, however, is the fiber-containing composite. The fibers may be either continuous-filament or woven. They might be short randomly-oriented fibers, they might be self-bonded to one another as well as to the matrix, or they might be true whiskers, of extremely small cross section and very high strength. Composite types include those made with ceramic fibers in either a metallic or a resin matrix, and those made with metal fibers in a ceramic matrix. More recently there has been attention to the possibility of using ceramic fibers in a ceramic matrix. Where continuous filaments are used, the fiber is wound on a mandrel of the proper type to give the desired shape to the piece and the matrix material is impregnated into the filament windings. Where the fibers are randomly oriented, the matrix material and fiber mixture may be formed by any of the conventional ceramic techniques.

In making a composite containing a fiber it is necessary first of all, of course, to have a fiber of high-tensile strength. If it is not woven into a free-standing structure, the fiber must be combined with a matrix material which will hold it in place and give the compressive strength necessary for the structure. The fiber and matrix material must not react unduly. There is a best amount of bond between the fiber and matrix so that the load can be transmitted throughout the piece. The fiber and matrix must have the correct relationship of thermal expansivities. In the fabrication of composites made with metal fibers and a ceramic matrix and those made with ceramic fibers and a metallic matrix, the problems of reactivity between matrix and fiber in the firing of the structure and the internal stresses of thermal expansion mismatches during the firing treatment are formidable. The same holds true, of course, for a composite made of a ceramic fiber in a ceramic matrix.

In structures made with filament-wound fibers and an impregnated matrix which requires no elevated heat treatment, the size and geometry limitation is not a critical one. In such a structure there is considerable latitude to form the flexible fibers onto a mandrel of any desired size and shape and then impregnate the woven fiber with matrix material. If a heat treatment of the matrix material is required, then the shrinkage and firing characteristics of the matrix impose size and geometry limitations. The limitations here are the same as for conventional fired ceramics. Similarly, the composites with randomly oriented fibers formed by conventional ceramic techniques have the same size and geometry limitations as conventional ceramics.

4. Bulk Placement and Molding

Ceramics may be formed to advantage by bulk placement or molding when a large or irregular shape is desired, or when the material is to be placed at the site of the utilization. This technique is also often used when it is desired to make a limited number of special shapes. Forms or molds can be made simply and cheaply since mold strength requirements are not stringent. The material may be cast into a mold, rammed, troweled, gunned, or poured like concrete into forms. The materials so formed may have a rigid structure provided by a hydraulic cement such as a high-alumina cement, or a chemical bond such as provided by sulfates, phosphates, chlorides, sulfites and silicates. Typical of these bonded materials are the refractory concretes, castables, and ramming mixes widely used in the heavy refractories industry. The materials might also merely be mechanically compacted to a rigid shape and develop a bonding phase only after heat treatment.

Typically, a mixture formed by this technique, taken in a broad sense, contains the sized aggregate which is the desired residual material or the end product, additives to enhance the formability, water to provide for rheological properties, and, depending upon the type of product, a cementing material.

The water content is critical, not only to the ease of forming or placement, but also to the density and strength of the product. Vibration is finding increasing use for better flow into complex molds, and for higher and more uniform density. Insulating materials of controlled density are also widely employed.

Because the forces applied in this forming process are comparatively low, any characteristics of the mixture being formed which influence the flowability become very important. Critical factors include besides the water content mentioned previously, size, shape, and surface activity of the aggregate grains, and amount and behavior of additives. The setting rate and strength of a cementitious phase is influenced greatly, of course, by the water content and the nature of the additives used.

Close control must be exercised over the rate at which water is removed from a formed piece. Cracking, or even explosion of massive pieces, particularly in job-placed products, may result if too-rapid water removal is attempted. The heat of hydration of high alumina cements may be large enough to require special provision for water cooling during placement, or large installations may have to be poured in carefully sized, alternate sections to avoid thermal rupture during curing. With these precautions this type of process lends itself to the forming of many sizes and shapes, particularly large ones, not obtainable by other forming techniques.

5. Impregnation

The technique of forming ceramic products by impregnation involves only chemical forces, not the usual mechanical forces of the more common forming processes. Impregnation may be considered a secondary forming process in that it is applied to a skeleton already shaped and sized by another technique. Commonly, impregnation is used to incorporate into a structure other elements or compounds which could not be so placed in the initial fabrication of the structure.

The infiltrant material function may be to fill pores, or to exert a bonding action, or to confer specific properties. In any case, the driving force for the impregnation is the surface free energy of the system. The impregnation is accomplished at temperatures, atmospheres, and pressures (both vacuum and high pressures) dictated by the chemical and physical properties of the impregnant and the skeleton.

Impregnation often is combined with a heat treatment to provide for a reaction between the skeleton and a liquid phase impregnant.

Impregnation techniques have been applied to a considerable extent to the fabrication of high-density graphites and of high-temperature resistant, special refractory ceramics and metals.

Since the driving forces are chemical and the combinations of skeleton and impregnant almost limitless, controls required and size and shape limitations are not well defined. They will be determined by specific situations. In situations where a reaction occurs between the skeleton and impregnant, the extent of the reaction and, if two or more phases are present, thermal expansivity mismatches will strongly influence the quality of the product.

6. Reaction Sintering

In forming ceramics by reaction sintering, a compound is formed of two elements which react exothermically and the heat of this thermitic reaction is used to cause the sintering of the mass. The components to be reacted plus a throttling agent (inert material) and a binder are mixed and compacted into the desired shape. The compact may be surrounded by a shrouding material. Heat is applied to bring the specimen up to ignition temperature. At the ignition temperature, the thermitic reaction proceeds and the heat generated sinters the compact.

To control the rate of the reaction, the throttling and shrouding material moderate the reaction and the furnace temperature is closely controlled. Controlled atmosphere also may be used.

In addition to the forming of solid articles by reaction sintering, attempts have been made to take advantage of the thermitic reaction to sinter a coating on a substrate which could not normally endure the required firing temperature for the coating. To date there has been limited success with such coatings because of adherence problems, but an area of potential usefulness appears to be indicated.

To make sound compacts by the reaction-sintering process, it is essential that all volatiles and chemical water be removed before ignition takes place. If not, disruptive forces and nonuniform temperatures will cause degradation of the compact.

Dimensional stability and control of reaction rates are problem areas. Specimen shapes must be kept simple. Compacts up to one inch in size have been reported.

7. High-Energy-Rate Forming

High-energy-rate forming processes have been developed in recent years and used primarily for forming of metals. Some exploratory work has been done to determine the usefulness of high-energy-rate forming processes for ceramics. Two types of high-energy-rate techniques have been used for the compaction of ceramic powders. In one, direct explosive charges are used, and in the other the compaction takes place in a pneumatic-mechanical press. In the direct explosive technique, the explosive charge is placed immediately around the material to be compacted and the energy dissipated outward is absorbed in a body of water. In the pneumatic-mechanical technique, a rapidly expanding gas propels a ram at high velocity (up to 60 feet per second) to the material to be compacted. Pressures applied have been calculated to be in the range of about 1-1/2 million to 7 million psi. In both the explosive-forming technique and the pneumatic-mechanical technique, the pressures generated are of sufficient magnitude to cause metals to behave plastically and flow. Some evidence of plastic deformation in ceramics has been obtained in exploratory work, but, to date, brittle behavior is the more common characteristic of ceramics exposed to the high-energy-rate forming.

In the explosive forming of ceramics, the amount of pressure and the rate at which the pressure is applied can be varied by controlling the placement, the amount, and the type of explosive charge used around the specimens. In the pneumatic-mechanical equipment, the pressure applied will depend, of course, upon the velocity and mass of the ram as it strikes the compact.

The response of a ceramic material to a high-energy-rate forming process will depend, of course, on the characteristics of the material. Some of the factors which affect the response are crystal size, crystal orientation to the shock wave, and the crystal structure. Also, of course, the mechanical characteristics of the material affects its response. An insufficient plastic flow of the material will cause it to be fractured. Elevated temperature operations would be expected to improve the response of many ceramics because of the greater likelihood of plastic flow, but little work has been done in this area.

The exploratory work which has been done with ceramics indicates that it is possible to increase the sinterability of ceramics by forming them under a high-energy-rate condition, allowing subsequent use of much lower sintering temperatures. Powder materials have been densified to as high as 95 percent of theoretical in high-energy-rate forming, but in most cases this appears to be a mechanical densification and no significant bond strength develops. In MgO, a coalescence of grains has been observed even though there was not an appreciable strength development. In quite highly densified compacts, there often are macrocracks, resulting presumably from the stresses caused by the reflection of shock waves after the initial densification. In a few selected ceramics there has been enough strength developed to indicate some promise that specimens could be formed to useable shapes in a high-energy-rate forming process without the subsequent sintering operation. At the present time, however, it appears that this method will be most useful in compacting materials to densities and structures not otherwise obtainable so that they sinter more readily. Some defect structures which would enhance sinterability have been noted.

The work with high-energy-rate forming of ceramics is still in an exploratory stage to the degree that it is difficult to set the size and geometry limitations. In the pneumatic-mechanical system, specimens with a 12-inch plan diameter are the maximum that can be made currently. The thickness limitation of such specimens has not yet been determined. A problem area exists in that spring-back of ceramic materials after the ram has struck and densified the compact will cause the compact to fracture.

In the forming of ceramic specimens using direct explosives, the cross-section is the limiting dimension at the present time. Quite long pieces can conceivably be made, but the difficulty of transmitting the energy through and to the center of a large cross-section piece has not been overcome to date. The cross-section limitation will depend upon the density of the material and its absorbing characteristics. With a very high specific-gravity material of a cross-section greater than two inches, sound compacts are difficult to produce. A lighter material might be formed in a larger cross-section.

When the ceramic work piece being formed has an appreciable mass there is a difficulty in releasing the load applied at a slow enough rate to prevent the specimens from fracturing. In the forming using explosives, the specimens can be contained within a metal can which will restrain them from cracking as the explosive energy dissipates. To date, the geometry has been limited to very simple shapes such as rods, plates, or hollow cylinders.

The uniformity of ceramics obtained from the explosive-compaction operation has not been good due to the variation in stress-wave strength as it progresses through a part. Often, internal fractures will occur at the center of the compact due to the tensile reflection of the shock waves as they meet at the center. In the pneumatic-mechanical process, the uniformity of compacts will depend on the amount of lateral flow which has occurred during the compaction.

8. Electrophoretic Forming

In forming ceramics by electrophoretic deposition it is necessary that the ceramic particles from which an article is to be made acquire an electrical charge. Usually this is accomplished by absorbing an ionic material onto the surface. The particles then are suspended in a low dielectric fluid such as alcohol in a system which provides for a voltage to be impressed across the suspension. Voltages might range from 100 to 1,000 volts. Under the influence of the impressed voltage, the ceramic particles will move and deposit on one electrode. The characteristics of the deposit will depend upon the particle charge carried, the size (usually about 60 microns and finer) and size distribution of the ceramic particles, the particle shape, and the voltage applied. If the particle size distribution is correct a dense layer can be formed. It is common practice to add to the suspension a material to cause the formed layer to have adequate green strength. Materials such as gelatins and polyvinyl alcohol are suitable binders.

If it is desired to use electrophoretic deposition to form a ceramic coating, the electrode can be the substrate and the product can be made by appropriate heat treatment of the coated substrate. If free-standing ceramics are desired the deposited material can be removed from the electrode and fired in a normal operation. The geometry of ceramics formed by this process thus is set by the geometry of the electrode. Ceramic depositions up to 1/2-inch thick appear feasible.

The electrophoretic techniques are well established and the fundamentals understood, but the potential usefulness of this forming method does not appear to have been established.

9. Machining and Grinding

Machining and grinding processes are used to give a final shape, dimension or surface finish to ceramics which are already shaped and fired. Two types are commonly used. One is the conventional grinding, using abrasives either in the wheel or powder form. Such grinding is used to provide the final form of the surfaces of the ceramic. Abrasive wheels also are used commonly for cut-off work. The other principal type of process for the machining and grinding of ceramics is termed ultrasonic machining, used primarily for holes, slots, and depressions of the ceramic. In general, machining and grinding operations are used to provide dimensional accuracy, complex designs not obtainable before final heat treatment, and control of the surface texture. Usually, machining and grinding are used only when no other forming process will provide these requirements to the degree needed.

In the abrasive grinding of ceramics, well-developed equipment and wheel designs are used. The abrasive used in most cases is either silicon carbide or diamond. Other abrasive grains rarely are used for the moderately hard ceramics. Silicon carbide is usually the choice for grinding all but the very hard ceramics. For the hardest ceramics, diamond abrasive is required. The abrasive must be harder than the work piece, with material removal depending somewhat on the difference in hardness between the abrasive and the work piece. The greater the difference the more rapid the material is removed. The silicon carbide abrasive is used in the loose form in a lapping operation, in a wheel with a vitreous or resin bond, or attached to a cloth or a paper belt. The diamond abrasive usually is bonded with metal on a metal wheel or lap.

A grinding operation generates heat, resulting sometimes in cracking due to thermal shock. For the protection of both the work piece and the abrasive, coolants are used. A considerable variety of coolants are available and most are satisfactory and are chosen primarily on the basis of shop preference.

The removal of ceramic material in a grinding operation is considered to be slow and expensive. Feeds and speeds are selected on the basis of experience and, of course, on the characteristics of the material being ground. Removal of more than 0.015 inch of material per pass is unusual. For good surface finish, removal rates often are limited to less than half of this amount.

The material removal and surface finish is influenced by the porosity, crystal size, and the structure of the ceramics being ground. Porosity, of course, can prevent the obtainment of a good finish. If there is more than one phase present and the phases differ in hardness, the hardness differences will affect the surface finish detrimentally.

The size limitations on surface grinding are not a particular concern if the surface to be ground is an exterior one. Interior surface grinding poses many problems if the length-to-cross-section ratio is great. In such cases, the usual practice is to machine ultrasonically rather than to use conventional abrasive grinding.

In abrasive grinding operations there is material fracture, chemical reaction between the abrasives and the ceramic work piece, and thermal shock. A better understanding of the phenomena which occur in grinding in all of these areas is required.

Ultrasonic machining is used to shape ceramics where abrasive grinding is not suitable. Basically, the ultrasonic machining technique uses a shaped tool and an abrasive slurry and impresses the tool image into the work. It is feasible to make a cut of any required shape by giving the tool the desired form. The tool is pressed against the work piece with a light pressure and an alternating motion. An abrasive slurry is fed between the tool and the work piece. The machining is accomplished by the chipping caused by the abrasive being hammered against the work piece by the vibrating tool. In such an operation it is essential that the work piece be brittle and the tool be tough and comparatively soft. The liquid containing the abrasive acts as a coolant, carries the wear debris away from the working face, and provides for an efficient transfer of energy from the tool to the abrasive and the work piece. Cavitation of the coolant liquid also is an essential feature of ultrasonic machining. The bubbles formed at the work face by the cavitation stirs the abrasive slurry and aids in removing chips of the ceramic being machined and of the worn abrasive.

The tool is given an oscillatory motion by an electrical transducer. Cutting rates are dependent upon the oscillatory amplitude, on the static load imposed on the tool, and on the size and nature of the abrasive particles. Diamond, silicon carbide and boron carbide abrasives are commonly used.

Cutting speeds will vary considerably, of course, because of the complex shapes that are machined by this method.

Since the abrasive grains must pass around the edges of the tool, the machined shape is always oversize relative to the tool. The oversize is a little greater than the dimensions of the abrasive and becomes less as the penetration goes deeper. The accuracy of the dimensions depends, also, of course, on the alignment and the stiffness of the tool. In operations using relatively coarse abrasive (200 mesh) the dimensions can be held to about 0.002 inch. Where greater precision is required and finer abrasive is used, dimensions as close as 0.0005 inch have been obtained.

For most of today's ceramics, ultrasonic machining can be used to produce almost any desired size and external geometry. Long holes are a problem, however, due to tool drift and to difficulties of maintaining the desired flow of abrasive slurry to the working face.

Ultrasonic machining is a relatively new process, and improvements in machine design are continuing.

III. FUNDAMENTALS OF FORMING

In deriving a critical picture of forming, the actions have been to first catalog the variety of methods which are being utilized or advocated. Then a classification has been presented so that the dozens of techniques could be analyzed in logical fashion. An attempt has been made to seek the similarities as well as the differences among the techniques, and establish common threads which weave through the diverse procedures and determine the physical structures realized.

The prospective common threads applying to forming identified so far are listed

(1) Characterization of the material feed.

(2) Characterization of the process as a controlled engineering system.

(3) Rheological response of materials to forming energy.

(4) Topological aspects of the packing of matter (space filling).

(5) Bonding and phase interaction.

(6) Forming stress interpretation.

(7) Product realization.

(1) Characterization of the material feed to the forming process has been discussed at length in earlier sections. It includes not only the raw materials themselves but also the prepared agglomerate mixture of materials.

(2) The phrase "Characterization of the process as a controlled engineering system" indicates the need for instrumentation which will measure the significant processing variables for each forming technique so that impersonal controls can be established. These should be developed to be as satisfactory for the forming process as temperature measurement and control have become for heat treatment. The factors which need to be detected and controlled will be learned from the science of deformation and flow of matter: rheology.

(3) Rheology evaluates the response of the material to the forming forces. This response has been discussed at some length in the section on cold forming, but is inherent in consideration of any forming technique. In every case the material which is being formed comprises a complex system, usually a three phase assemblage of solid, liquid, and air.

Inadequate understanding and poor control of the rheology of the material being formed can initiate flaws which persist through to the finished product. This well illustrates the need for repeated communication between the scientist accumulating fundamental understanding of the phenomena and the engineer exploiting the phenomena to make a useful product. For example, although ceramic engineers have utilized an understanding of rheology of suspensions in slip casting with success, there still remains a large body of knowledge in this regard that could be profitably exploited. Rheolcgical properties of dry powders are a fascinating field for research and process development which have scarcely been touched.

(4) Space filling involves the geometrical considerations which govern the packing of matter whether atoms, individual grains from submicron to the coarsest screen sizes, or assemblages of particles.

Accompanying forming is a densification by packing of particles or granules. The scientist investigates how this compaction occurs: whether by sliding of particles past each other; by elastic, plastic, or viscous deformation; by fracture of particles; or by diffusion of atoms across phase and grain boundaries.

(5) Always accompanying the geometrical phenomena of packing are the force phenomena which account for the bonding strength in the impressed shape. Here the scientists argue over whether the strength of a cold-formed piece arises solely from mechanical interlocking, or adhesive phenomena, or actual particle to particle welding by a chemical interaction. Hot forming exemplifies the attempt to simultaneously achieve packing and bonding while controlling grain growth.

(6) Forming processes may impose two types of stresses in ceramic shapes. First the thermal, liquid, or pressure gradients that may exist in shapes during forming may create long range stress distributions or body stresses. These stresses may lead to warpage or cracking of the ceramic shape. The effect of these body stresses on the resultant properties of the formed shape are difficult to assess because of the relatively few methods available to detect and measure them.

In addition to body stresses, microstresses on and between individual grains may be developed by the difference in physical properties of different phases or the anisotropic character of a single crystalline phase. The ultimate of microstresses effect on the mechanical properties of a ceramic are still open to question even though the techniques for their determination are more advanced than those available for body stress analysis.

(7) "Production realization" encompasses the whole range of technical, economic, and strategic considerations which arise from the fact that the processing of ceramics must result in quality products for which someone is willing to pay. The products must not only be controlled as to structure and properties, meet dimensional and surface finish

specifications, and be free of flaws, but also must be competitive. Availability may be of prime strategic and economic importance.

Realizing all this will always demand considerable skill on the part of the processor, but again a progression of "characterization" of the material in process and of the final product is proposed as an essential requirement.

During the course of this survey it became evident that semantic clarification required a distinction between the terms "structure" and "microstructure." Microstructure has been taken here to mean the qualitative or quantitative description of the relationships between phases present. This is true whether seen with the unaided eye, with a light or electron microscope, or with other analytical tools for extending resolution. Structure is intended to mean the total description including microstructure, chemical and mineral composition, purity, and submicroscopic features relating to imperfections. The attainment of a controlled, reproducible structure is the initial objective of the forming process.

"Characterization" implies not only knowing the structure and selected physical properties at each stage of processing, but knowing also how all these correlate. As we improve our ability to add this knowledge of characterization to engineering judgment, the accomplishment of the goals of processing occurs faster, cheaper, and more effectively.

IV. SUMMARY AND CONCLUSIONS

The state of the art of the forming of ceramics has been surveyed within the limitations imposed by a tight time schedule of only four months. Information was sought both by a search of the literature and by personal interviews through group conferences and individual contacts. The results are presented in individual reports on the topics: (A) characterization of particulate systems, (B) cold forming, (C) hot forming, (D) melt forming, and (E) miscellaneous forming processes, as well as in two bibliographies based on a specially developed classification scheme.

It is evident from the range of the reports and the references that forming, as one step in the total processing sequence, is undergoing a dynamic expansion of attention and competence in research, development, engineering, and manufacturing. Specialized leadership has been contributed by the nuclear fuel industry because of its particular end use requirements and economics. The pioneering of the aerospace industries is also beginning to become apparent, while the more traditional and established portions of the ceramic industry have certainly not been timid about introducing timely innovations and improvements.

Needs for the future appear to include a unifying of the technology by a recognition of the common elements in the diverse forming methods, and a closer interaction between scientists and engineers for the mutual stimulation of both.

It is not feasible to attempt to select the most promising forming methods for making a particular material in a specific shape. The selection must be based on a complex of considerations of end use and service environment, shape, size, design, tolerances, time available, and money available, so that each case must be individually appraised. It is probably true that of the many forming processes here examined, none will ever be wholly discarded or exclusive. Each has a special contribution to offer to solve a specific problem in forming.

V. BIBLIOGRAPHIES

1. Procedures

Concerning the bibliography on "Methods for Forming Ceramics" the following comments can be made on method and completeness. In the time available the intensive search for references has been pretty much limited to the years 1954 through July 1963. The search has been largely based on Ceramic Abstracts and Nuclear Science Abstracts for the sections on forming, and on Chemical Abstracts (1958 through July 1963) for the section on characterization of particulates. Spot checks were made in the abstracts section of Transactions of the British Ceramic Society and OTS Reports (Office of Technical Services, U. S. Department of Commerce). Certain outstanding papers were pursued after being cited as footnote references.

Though the bibliography is necessarily incomplete, it is extensive and representative. As it happens the years covered have witnessed a high level of activity in the field of forming of ceramics, led and dominated by the nuclear field.

The collected and selected references are presented in the bibliography which follows. This bibliography has been gleaned from a more extensive and detailed set of references, complete with abstracts, which are mounted on ASM-SLA Metallurgical Punched Cards. One complete set of these cards has been submitted with this report to the contracting agency. A second set has been retained at the Department of Mineral Technology, University of California, Berkeley. They can form the nucleus of a continuing and more extensive bibliography on the topics treated, and also as the basis for coverage of other topics in Ceramics.

Any reader who misses certain favorite references in the area of his special knowledge is welcome to call attention to them for future expansion of this bibliography.

It would have been desirable to have included a brief, critical description of each of the references cited, in the context in which it has been classified. This has not been feasible in the time available for this initial survey. It is hoped that it can be done in any continuations.

2. Storage and Retrieval of Technical Literature

As the number of references accumulates in a bibliography or any information storage system, it becomes necessary to settle upon some classification scheme. Information storage and retrieval, also called documentation, is at present in a period of rapid growth and change. A variety of coding techniques is being advanced. It is best to agree that there is no one best way, but the system chosen must be individually determined for the amount and type of information being handled and the needs and personalities of the people being served.

For the purposes of the present bibliography it was determined to base the presentation on the classification and related edge punched cards adopted by the American Society of Metals, called the ASM-SLA Metallurgical Literature Classification, International (Second) Edition as revised in 1958.

The positive reasons for adopting this system were that it had been worked out in detail for the needs of metallurgists. In view of the trend in the government, industry, and universities to a unification of metals, ceramics, and polymers into materials science and materials engineering, it would be interesting to see how few modifications would adapt the classification and card for Ceramics, starting with the limited and specialized field of "Methods for Forming Ceramics."

Though it was recognized that the most recent documentation practice stresses certain shortcomings in both classifications and edge punched cards, these do not enter during the early stages of documenting a new subject when the number of items is less than two or three thousand. As the number of items increases, it is feasible to shift to the so-called "peek-a-boo" type of cards, building on rather than abandoning the established classification and card file. Above twenty to thirty thousand items it first becomes necessary to think in terms of more advanced systems and computers. And even here the work done on the original classification and cards would still have served a useful function.

The classification coding scheme which has evolved is summarized in the following pages.

In organizing the bibliography an attempt was always made to keep it as simple as possible. Though the classification extends to fourth order breakdowns, references were grouped at the highest order which was feasible for the number of references to be scanned. That is, no groupings have been attempted at higher than third orders, and wherever reasonable the breakdown was stopped at second orders.

Inevitably there will be overlaps and repetitions in the use of classification. Various devices have been used to minimize these. Certain references appear under more than one topic, but attempt was made to classify each reference primarily under the topic which appeared to best describe its major and novel features. To a limited extent cross-references have been indicated by repeating references at two or more places in the bibliography. General review articles and books have been classified under general headings, rather than attempting to repeat them under each process mentioned.

Selected References on Documentation

ASM-SLA Metallurgical Literature Classification, International Second Edition
American Society for Metals, Cleveland 3, Ohio (1958) 74p. $3.00

Workbook for above in loose-leaf $5.00

ASM-SLA edge-punched cards and accessories may be purchased from:
E-Z Sort Systems--Vaughn Agency
236 North Clark St.
Chicago 1, Illinois
or
E-Z Sort Systems, Ltd.
45 Second St.
San Francisco 5, California

Information and supplies about "peek-a-boo" cards and related accessories in their "Thermatrex" system can be obtained from:
Jonker Business Machines, Inc.
Gaithersburg, Maryland,
or at four regional sales offices.

Among the many papers appearing on the topic of information systems, the following have been found useful:
Eugene Wall
Information Systems
Chemical Engineering Progress 55 (1) 55-9 (1959)

John W. Murdock and G. B. Simpson, Jr. (Battelle)
Practical Information Handling
Industrial Research, April-May 1961

Ralph Cushing
Improving Personal Filing Systems
Chemical Engineering, January 7, 1963, p. 73-88
Attention is called to:
Chemical Engineering Thesaurus
American Institute of Chemical Engineers
345 East 47th St.
New York 17, New York
$10.00 to non-members

Thesaurus of all engineering terms
Engineers Joint Council
To be published in 1964

National Bureau of Standards
Technical Note 157
Bibliography on Coding Systems

Bart Holm
Coordinate indexing
Engineers Joint Council $1.00

Literature and training courses available from Battelle Memorial Institute on information handling

Acknowledgements for discussions in information handling:
Ralph L. Darby, Battelle Memorial Institute
R. S. Sheldon, American Ceramic Society
E. Dugger, Research and Technology Division, U. S. Air Force.

3. Coding Scheme Adopted for These Bibliographies

The detailed classification scheme which has been followed in preparing the two bibliographies included in this report is presented in the following six pages.

Classification Scheme Adopted for Current Purposes

Based on ASM-SLA Metallurgical Literature Classification
International (Second) Edition (1958)

Processes and Properties Schedule supplemented by
Products and Equipment Schedule

First Order Divisions

A - General
B - Ores and Raw Materials
  Geology and Mineralogy
C - Batch Preparation
D - (Iron and Steelmaking) - (tentatively retained)
E - Melting. Foundry
* F - Primary Forming
* G - Secondary Forming and Surface Finishing
  Machining and Grinding
* H - Particle Technology
    Powders, Fibers, Filaments, Whiskers,
    Sheets and Films
J - Heat Treatment
    Including Sources of Heat, Fuels and
    Combustion
K - Assembling and Joining
L - Cleaning, Coating and Finishing
M - Microscopy, Constitution and Primary Structure
N - Transformations and Resulting Structures
P - Physics
    Physical Properties and Their Measurement
Q - Mechanical Properties and Tests
R - Chemistry
    Chemical Properties
    Corrosion
S - Inspection and Control
  Measurements - Metrology
U - Allied Fields
T - Products and Parts
* W - Plant Equipment
    Production Equipment and Layout
    Unit Operations
X - Laboratory and Control Equipment

*Principle categories used in this bibliography

Second Order Divisions

F - Primary Forming
  Molding. Compacting. Shaping.
    Consolidation

1. Cold forming from "dry" powders
2. Cold forming from pastes (plastic masses)
3. Cold forming from suspensions
4. Polymer-assisted forming
5. Chemically-assisted forming
6. X (Forbidden)
7. Forming by combinations of energy forms
8. High energy rate forming
9. Open
10. Forming glasses
11. Fusion casting
12. Hot deposition
13. Open
14. Open
15. Forming specific shapes
16. Forming porous bodies--foaming
17. Forming composites
18. Open
19. Open
20. Theoretical bases of forming
21. Preparations for forming (linked to C-Batch Preparation)
22. Open
23. Hot forming
24. Open
25. Vapor forming
26. Forming from filaments or films
    Related to F10g
27. Forming from solutions; electroforming
28. Open
29. Forming - general

Third Order Divisions

F - Primary Forming

1. Cold forming from "dry" powders
   - a. General
   - b. Rigid die pressing
   - c. Flexible die pressing
     Isostatic, hydrostatic; polymeric die liners
   - d. Pressing in evacuated dies; vacuum-assisted
   - e. Extruding
   - f. Dieless compacting; powder rolling
   - g. Vibratory compacting; jolting
   - h. Impact forming
   - j. Dry process enameling
   - m. Superpressure forming (above 100,000 psi)
   - n. Zero pressure forming; flow forming
   - p. Supplementary energy to pressure (mechanical)
   - r. Rheological controls and behavior
     Response of material
   - Absorptive die pressing - see F2d
   - Additives - see F21s
   - Design of parts - see F21k

2. Cold forming from pastes (plastic forming)
   - a. General
   - b. Manual
   - c. Pressing
   - d. Pressing - absorptive mold
   - e. Extruding (clay-water type)
   - f. Dropping, impacting
   - g. Turning (jiggering, jollying, "flower pot" forming
     Linked to G2
   - h. Tamping, ramming
   - j. Cements, trowelling
   - k. Cements, gunning
   - m. Silk screening
   - n. Rolling, coiling
   - p. Supplementary energy to mechanical
   - r. Rheological controls and behavior
     Response of material
   - s. Steam - assisted; warm working
     (Also see F23k)
   - Additives - see F21s
   - Mechanization, Automation - see W24

3. Cold forming from suspensions
   - a. General
   - b. Slipcasting, gravity and general
   - c. Slipcasting, superimposed motions
     (vibratory, centrifugal)
   - e. Slipcasting, vacuum or pressure assisted
   - g. Cement casting
   - h. Surface coating from suspensions:
     porcelain enameling, glazing, etc.
   - j. Application techniques: brushing, spraying, dipping, slush coating, electrostatic spraying
   - p. Supplementary energy to mechanical
   - r. Rheological controls; additives (see also F21s)
     Response and behavior or material
   - Molds - see F21g
   - Design of molds - see F21j
   - Additives for specific purposes - see F21s

4. Polymer - assisted forming
   - a. General
   - b. Compression molding
   - c. Transfer molding - thermosetting
   - d. Injection molding - thermoplastic
   - e. Extrusion
   - f. Rolling
   - g. Casting (Hot spray, . .)
   - h. Vibratory-assisted
   - j. Vacuum-assisted or Blown
   - r. Rheological controls and behavior
     Response of material
   - s. Binder-plasticizer (polymer) selection and content (see also F21s)
   - Additives for specific purposes - see F21s

5. Chemically - assisted forming
   - a. General
   - b. Cement setting
   - g. Reaction sintering; reaction pressing

8. High energy rate forming
   - a. General
   - b. Explosive forming
   - c. Spark discharge
   - d. Ultrasonic impulse

10. Forming Glass
    Linked to E-Melting, Foundry
    - a. General
    - b. Pressing
    - c. Blowing
    - d. Combinations of pressing and blowing
    - e. Extruding
    - f. Drawing
    - g. Fiberizing
    - h. Dropping, bending
    - m. Preparations for forming: gathering, feeding, marvering, forming blank or parison (see also F21)
    - n. Casting: free flow, centrifugal, injection
    - p. Rolling
    - r. Rheological controls
      - Dies and Molds - see F21g
      - Porcelain enamel application - see F1j, F3h, F3j
      - Forming specific shapes (marbles, plates, etc.) -- see F15

11. Forming by Melting and solidification of non-glassy ceramics in bulk (Fusion casting).
    Linked to E-Melting, Foundry
    Also see F10, F12
    - a. General
    - b. Pouring techniques
    - c. Cooling in place
    - d. Molding techniques (see also F21g, h)
    - e. Continuous casting
    - f. Single crystal growing
    - r. Rheology; Response of material

12. Forming by hot deposition - Melting particles individually
    - g. Flame spraying
    - j. Arc spraying
    - k. Plasma arc spraying
    - m. Detonation (explosive propulsion)
    - r. Response of material
    - s. Source of material

15. Forming specific shapes
    - a. General
    - b. Single crystals (see F11f; . . .)
    - c. Cylinders, rings
    - d. Cones, ogives
    - g. Sheets, plates, films
    - j. Laminates, casing
    - k. Spheres, pellets, marbles
    - m. Hollow-ware incl. crucibles, boats
    - n. Rods and bars
    - p. Tubing
    - q. Complex designs
      - Fibers - see F10g

16. Forming porous bodies - foaming
    - a. General
    - b. By underfiring
    - d. Honeycombing
    - g. Light-weight aggregates; bloating
    - n. From powders by gas evolution
    - p. From melts by gas evolution
      - Gas opacified enamels and glasses

17. Forming composites
    - a. General
    - b. Reinforced structures, forming of
      - Impregnation; infiltration see H16d, e

20. Theoretical bases of forming
    - a. General
    - b. Applied mechanics of (see F21j, k)
    - d. Applied rheology (see F21r, F1r, F2r, F3r, F10r, etc.)
    - f. Theory of space filling - compaction

21. Preparations for forming
    Linked to C-Batch Preparation
    - a. General
    - b. De-airing
    - c. Granulation
    - d. Coatings on granules
    - f. Feeding
    - g. Dies and molds
    - j. Design of dies and molds
    - k. Design of parts
    - r. Adjustment of rheology (see F1r, F2r, F3r, F4r, F23r)
    - s. Additives for specific purposes

23. Hot forming (pressure sintering)
    - a. General
    - b. Forging
    - c. Rolling
    - d. Drawing; i.e. wire drawing
    - e. Extrusion
    - g. Coining or stamping
    - h. Swaging; impact forming (see also F1h)
    - k. "Warm" pressing (see also F2s)
    - m. Zero pressure applied
    - n. Pressing - atmospheric and general
    - p. Pressing - vacuum
    - q. Pressing - isostatic (pressure bonding)
    - r. Response of material; flow; rheology
      - Dies and molds - see F21g
    - Reaction pressing - see F5g

25. Vapor forming (gas plating, vapor plating, vapor deposition)
    - a. General
    - b. Co-deposition
    - g. Physical - general
    - h. Physical - vacuum evaporation
    - j. Physical - very high temperature heating
    - k. Physical - sputtering
    - m. Chemical - general
    - n. Chemical - thermal decomposition (pyrolysis, surface - catalyzed)
    - p. Chemical - hydrolysis
    - q. Chemical - reduction by hydrogen
    - r. Chemical - reduction by carbon
    - s. Chemical - oxidation

    Coating of granules - see F21d

26. Forming from films, filaments, or whiskers
    Linked to F10g
    - a. General
    - b. Felting
    - c. Weaving
    - d. Filament winding
    - e. Polymer coated, bonded, or impregnated
      - See for forming of initial films, filaments, or whiskers F10g, F11f, etc.

27. Forming from solution; electroforming
    - a. General
    - b. Electrophoretic (cataphoretic)
    - c. Anodization
    - d. Electroplating

29. Forming - general

Second Order Divisions

G - Secondary Forming and Surface Finishing
  1. Secondary Forming (Machining) Operations in general
  2. Forming from "green" (unfired) blanks--general
     Linked to F2g
  3. Forming from partially fired blanks--general
  4. Forming from fully matured ceramics--general

  8. Manual operations

  15. Chemical forming, surface finishing, and etching
  16. Mechanical operations--stamping and punching
  17. Mechanical operations--machining by cutting action
  18. Mechanical operations--grinding
  19. Mechanical operations--surface finishing
  20. Mechanical operations--impact and abrasive blasting
  21. Mechanical operations--sawing, cutting
  22. Thermal operations--forming, cutting, surface finishing, etching
  23. Final forming
  24. Forming by combinations of energies--special material removal methods
  25. Applied mechanics of forming and machining
      Stress analysis
     Measurements, Metrology (see S)

Third Order Divisions

G - Secondary Forming
15. Chemical processes
    h. Material removal - "chemical machining"
    j. Etching
    k. Polishing

16. Mechanical operations - stamping, punching
    n. Stamping
    p. Punching
    q. Coining
    r. Embossing and Engraving

17. Mechanical operations - machining
    a. Turning
    b. Milling and hobbing
    c. Planing and shaping and facing
    d. Boring and broaching
    e. Drilling and reaming
    f. Tapping and threading
    g. Gear cutting and screw cutting
    j. Filing
    k. Machinability
       Sawing - see G21
       Fettling - see G2

18. Mechanical operations - Grinding
    a. General
    b. Barrel rolling
    e. Vibratory - assisted (see G24c)
    g. Cylindrical
    h. Centerless
    j. Internal
    k. Surface
    m. Form grinding
    n. Grinding with loose abrasives
    p. Grinding holes, core bits
    r. Diamond tooling

19. Mechanical operations - surface finishing
    a. General
    b. Barrel burnishing; tumbling
    c. Buffing
    e. Vibratory - assisted (see G24c)
    n. Honing
    p. Lapping
    q. Superfinishing
    r. Other
    s. Polishing with loose fine abrasives

20. Mechanical operations - impact, abrasive blasting

21. Mechanical operations - sawing, cutting
    a. Contour
    b. cut-off
    c. Wire cutting

22. Thermal operations
    g. Flame processes - reshaping, "lampworking"
    h. Processes utilizing very high temperature heat sources
    j. Material removal
    k. Surface finishing - fire polishing
    m. Thermal etching
    n. Cutting
    p. Drilling holes

24. Forming by combinations of energies
    a. Spark
    b. Chemical-mechanical (see also G15)
    c. Ultrasonic, electromechanical
    d. Electrochemical
    e. Arc (see also G22h)
    f. Magnetic impulse

Second Order Divisions

H - Particle Technology
    Powders, Fibers, Filaments, Whiskers,
        Sheets and Films
10. Particle powder preparation
11. Properties of particles
12. Properties of assemblies of particles
13. Mixing and blending
    Related to C
14. Granulation, agglomeration
    Related to C, F21c
15. Sintering
    Related to J
16. Secondary operations on porous bodies
17. Fiber Metallurgy (see also F17)

Third Order Divisions

H - Particle Technology
10. Particle-powder preparation
    a. Wet-chemical
    b. Electrochemical, electrical
    c. Comminution
    d. Decomposition and calcination
    e. Vapor-solid reactions
    f. Melting and solid state reactions
    g. Purification

11. Properties of particles
    a. Size, shape, density
    b. Surface area, gas inclusions
    c. Electrical, magnetic, thermal
    d. Impurity analysis, distribution of pores, crystal structure, defect
    e. Solid-liquid interface; Solid gas interface
    g. Reactivity of particle; relation to defects

12. Properties of assemblies of particles
    a. Size distribution
    b. Particle packing, packing density, tap density
    c. Flow, angle of repose
    d. Rheology of solid-fluid systems
    f. Permeability

13. Mixing and blending
    a. Mixing theory
    b. Dry mixing
    c. Wet mixing

14. Granulation, agglomeration
    a. Pelletizing
    b. Briquetting
    c. Green strength and compressibility
        Slip casting; Extrusion (see F1c; F1e; F3e; F4e)

15. Sintering (see J)
    c. Properties of sintered material; characterizing particles and assemblies of particles

16. Secondary operations on porous bodies
    d. Impregnation
    e. Infiltration

Second Order Divisions

W - Production Plant Equipment and Layout
1. Mechanical assembling equipment
2. Mechanical cleaning and finishing
3. Chemical and electrochemical cleaning and finishing
4. Other cleaning and finishing equipment

* 10. Plant installations
11. Power generation and distribution
12. Materials handling equipment
    Packaging and shipping
13. General service and maintenance equipment
14. Open
15. Ore and raw material preparation
16. Fuels
17. Calcining furnaces
18. Melting furnaces
19. Casting equipment

* 24. Forming and shaping equipment
* 25. Machine tools
* 26. Powder processing equipment
27. Heat treating furnaces and kilns
28. Other heat treating equipment
29. Joining and assembling equipment--
    welding, metalizing, brazing, soldering

*Most pertinent to present bibliography

Third Order Divisions

W - Plant Equipment
24. Forming and Shaping
    g. Presses
    m. Shears and saws
    n. Dies (see also F21g)
    p. Mandrels and punches

25. Machine tools (see G)
    a. Cut-off machines
    b. Jigs and fixtures
    c. Grinding belts and wheels
    d. Accessories
    n. Lathes
    p. Drills
    q. Shapers
    r. Millers
    s. Grinders

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(See also H12a)

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H11b. SURFACE AREA

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(also see F15g)

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APPENDIX

Recommended Study Areas to Further Ceramic Forming Processes

All authors have indicated need for development or research in their respective sections as part of their review because the critical surveys revealed lack or even complete deficiency of knowledge in many areas. Most of these ideas relate to bridging the gap between ceramic science and the engineering technology of processing. Many of them relate to intuitive, empirical or even proprietary thoughts as well as to the deductive thoughts that result from any attempt to correlate fundamentals with a highly technological subject. These recommended areas of study have been included as an appendix to this report and are paraphrased below.

(1) Critically delineate properties of particulate systems which are specifically related to forming theory. The importance of these properties to forming methods cannot be overemphasized.

(2) Determine relationships of both the bulk and surface reactivity of powders to such factors as particle size, shape, defect structure, stoichimetric composition, and atmosphere.

(3) Relate the flow properties of particulate systems to their shape and size distributions, reactivity, and particle-particle interaction. Study fluid-solid interfacial reactions.

(4) Develop methods and conditions for reproducing size and distributions of powders in the range of 15 microns and below, including methods other than mechanical comminution. For the different production methods, study the factors that determine the size, shape, and size distribution of resulting particles.

(5) Show the application of surface and organic chemistry fundamentals to the understanding of the function of organic additives in model particulate systems.

(6) Study the fundamentals of compaction response in particulate systems under high pressures at room temperature.

(7) Define the mechanism of compaction in particulate systems under programmed heat-pressure-atmosphere conditions.

(8) Study the interaction of ultrasonic energy and particulate systems during compaction processes.

(9) Support empirical development of ultrasonic energy aids to forming.

(10) Develop relations between forming energy, compaction response and extrusion rates in extrusion forming systems, hot and cold.

(11) Apply control theory to develop a sound basis for transmission of forming energy in forming operations treated as true engineering systems, hot and cold.

(12) Bring hot pressing processes from the laboratory to the production stage by engineering development.

(13) Develop novel techniques for heat treatment requiring low capital investment.

(14) Analyze simultaneous viscous flow and heat transfer in glass forming processes.

(15) Develop processes for utilizing glasses which either (a) are highly refractory, or (b) have viscosities substantially lower than conventional glasses.

(16) Continue the development of the theory of crystal growth and nucleation of glasses as related to forming ceramics.

(17) Develop glass forming processes which preserve the inherent strength of glass.

(18) Study the theory of crystal growth and nucleation of melts as a means of improving process operation and characterization of products produced by fusion casting.

(19) Develop application of thermodynamic data on ceramic oxide melts to fusion casting.

(20) Systematically study the material and operational variables as related to the characterization of shapes produced by spraying of matter or semi-matter particulate systems.

(21) Design transport devices for feeding submicron particle systems or agglomerates in hot spraying and other forming systems.

(22) Study the effect of impingement and configuration on mass and heat transfer during vapor deposition.

(23) Initiate a fundamental study of nucleation and crystal growth from the vapor state.

(24) Systematically review work on fiber ceramics and continue support of work to optimize the process to utilize fiber strengths in composite systems particularly for high temperature use.

(25) Study the mechanism of material removal in finishing, drilling, or shaping ceramics.