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BACKGROUND MATERIAL FOR SEMINAR ON

"THE THEORY OF SINTERING AND THE EFFECTS OF ANISOTROPY OF SURFACE AND GRAIN BOUNDARY ENERGIES ON SINTERING"

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SINTERING OF CERAMICS

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INTRODUCTION

The primary goal of sintering research is the controlled manipulation of microstructure. Out of the entire range of microstructures which are theoretically possible, each material system will be able to achieve only a subset of them, depending on the intrinsic material properties. Within these material constraints, the aim is to produce microstructures which enhance specific properties. Our understanding of the relationships among materials processing, microstructure, and properties is just beginning to emerge, and is producing unexpected results. For example, in a recent study of toughness in Al_2O_3 by Bennison and Lawn, microstructures with platy grains and a bimodal grain size distribution in undoped Al_2O_3 exhibited a greater resistance to crack propagation than did the more uniform microstructures in MgO-doped Al_2O_3 [1]. As a result of this emerging understanding, the focus of sintering science is changing from the modification of microstructures in incremental ways for correspondingly incremental improvement in properties to more effectual manipulation of microstructures to optimize properties. However, the production of the optimum microstructure will be dependent on both the material and the application and may require radically different processing routes for different materials. In this review paper, we have examined the research in sintering science over the past five years which has advanced the goal of microstructure manipulation.

THEORETICAL MODELLING AND MODEL MICROSTRUCTURES

Over the past five years, substantial progress has been made in developing more realistic sintering models and in designing materials to test these theories. The basis of the current theoretical advancement is that the specific set of coupled kinetic processes operating in certain systems can be separated and, thus, the enthusiasm for the modelling of competing mechanisms in sintering has been restored. When the sintering of a system cannot be categorized by a small subset of possible sintering processes, simplified geometries can be used to examine the roles of various processes in microstructure development. The recent advances in sintering experiments have been made possible by a revolution in the control of powder characteristics and processing. This revolution, fomented by H. K. Bowen, provides enormous flexibility in the design of specific microstructures [2]. In the following discussion, theories and their complementary model experiments are discussed together.

The simplest model whose use continues to provide insight is the two-sphere model. An example of recent results is the numerical simulation of neck growth by surface and grain boundary diffusion [3]. In agreement with previous studies [4], there exists a limiting ratio of grain boundary diffusivity to surface diffusivity above which the densification rate does not increase because surface diffusion is required to redistribute matter in the region of the neck.

The next step in geometrical complexity, is the sintering of a row of spheres. For this geometry, the number of concurrent processes which can be treated is large because each particle has only two neighbors and, if is assumed that there are no torques on the particles to rotate them out of the axially symmetrical position, the mathematical statement of the problem is simplified. An important new development is that differential densification has now been treated in a row of spheres by allowing a section of the chain to begin to densify before the rest of the chain [5,6]. It has been calculated that, when the other sections begin to densify the chain will either become uniform in its sintering or will break up catastrophically into individual segments depending on the relative rates of coarsening and densification and the dihedral angle.

This problem of differential densification and development of heterogeneous microstructures has recently been a topic of intense theoretical and experimental investigation [7-15]. The approach has been from two extremes in initial microstructures. In the first, mono-sized powders are packed and sintered, and the size of final defects are correlated with the initial amount of ordering [12,13]. In the second case, dense inclusions are placed into a powder compact and the effect of the non-densifying inclusions on the densification rate of the matrix is evaluated [14]. These two approaches reflect the extremes seen in the technology of powder processing with controlled composition and physical characteristics and of processing of ceramic-matrix composites. The types of heterogeneities examined over the last 5 years range from large cracks between regions of perfect packing to large spherical pores in a matrix containing smaller pores, to sintering of a porous matrix around a higher density spherical inclusion. The advances in sintering of composites are summarized in the next section.

A major result is that the maximum sintering stress is on the order of 0.1 - 2 MPa regardless of microstructure [7-11]. This result is important because the sintering stress determines the opposing tensile stress required to keep particles from densifying or, for higher tensile stresses, to pull particles apart. The size of the sintering stress is a major factor in specifying HIP'ing and hot pressing conditions to improve reliability of sintered composites.

Experiments on 2-D arrays of monosized or bimodally-sized particles have shown that crack-like defects develop in the imperfectly packed regions separating perfectly packed regions [12,13]. Liniger and Raj found that packing should be random to maximize final density and minimize final defect size. An additional important result is that differential densification between poorly packed and well-packed regions lead to the desintering of particle-particle necks, as predicted by the calculation for the row of sintering spheres. In addition, differential densification and desintering were found to be more important than asymmetrical neck growth in producing heterogeneous microstructure. The final result of this study is the suggestion that a *narrow but not mono-sized* particle size distribution provides the most homogeneous powder compact, the highest final density, and the lowest amount of sintering damage in the final sintered body.

As a result of the improvements in processing, primarily from the control of particle size and use of colloidal techniques, model uniform microstructures have now been fabricated in Al_2O_3 [16-19] ZrO_2 [20], Y_2O_3 [21,22], TiO_2[23,24], Si_3N_4 [25], and mullite [26,27]. In 1981, Rhodes' study of agglomerate effects on ZrO_2 sintering demonstrated that colloidal processing can be used to produce uniform, dense microstructures at low temperatures that show a resistance to discontinuous grain growth. A recent example is the use of colloidal processing of Al_2O_3 with a fine particle size (0.16 μm) and an extremely narrow particle size distribution to sinter Al_2O_3 to 99.5% of theoretical density at 1150°C [16,17].

The use of colloidal processing techniques for easily-sinterable materials, such as Al_2O_3 and TiO_2, creates microstructures which approach the simple geometrical models for sintering. With correct processing, microstructural parameters are representative of the microstructure at the scale of a few grains as well as the compact as a whole. These experimental developments permit a re-evaluation of 3-D sintering models based on uniform microstructures described by repeating unit cells [18,28-30]. The unit cell typically consists of a single grain of a space-filling geometry with pores along the boundary between grains. With these unit cell models, microstructure evolution during sintering is followed with the following features: competition between coarsening and densification and scaling laws for the sintering of a bimodal pore size distribution. The microstructural parameters needed experimentally to compare with the models are easily measurable parameters: density, average pore size, pore size distribution, grain size, grain size distribution, surface area, and so on.

There are many combinations of microstructural features that can be used to follow the competition between densification and coarsening, for example, pore size-grain size or surface area-boundary area [30,32-34]. The most reliably and easily measured are grain size and density. From plots of density versus grain size, changes in the slope of trajectories are due to changes in the ratio of coarsening rate to densification rate. These plots are of special utility because transitions between processes can be seen from changes in the slope for a given run and changes in microstructure with processing can be determined by comparing grain size at a different density [18,35]. For example, studies by Edelson and Glaeser and by Barringer showed that monosized TiO_2 follows the same density-grain size trajectory for the temperature range 1000° to 1160°C for a variety of processing conditions [23,24].

Systems which coarsen more than they densify have also been examined: TiO_2 in HCl [36], ZrO_2 in HCl [37], ZnO in H_2 [38], Fe_2O_3 [39]. Because each of these systems can be sintered close to theoretical density in air, the ratio of the coarsening rate to the densification rate can be varied by changes in atmosphere [40,41]. Studies of coarsening are important to our understanding of the generation of crack-like flaws by sintering. Specifically, coarsening processes maintain particle coordination without the generation of tensile stresses at particle contacts. When densification occurs, particles with lower coordination numbers will be under a tensile stress and these sintered necks will dedensify. If the necks sizes are increased by coarsening, dedensification and the generation of crack-like pores can be suppressed. These concepts have been demonstrated in studies of ZnO powders [42] and glass, alumina, and ZnO powders sintered to rigid substrates in which controlled amounts of coarsening at low temperature prior to high temperature densification led to denser final microstructures than with high temperature densification alone [43]. Grain growth during the intermediate stage of sintering has also been shown to increase homogeneity in pore size in a powder compact containing large pores by coalescence of smaller pores, while the larger pores grow less [44].

An additional refinement in sintering theory has been a consideration of how the free surface-grain boundary dihedral angle affects: the driving force for sintering [5,6,45], the breakup of cylindrical channels in the transition of intermediate to final stage sintering [46], the attachment of pores to grain boundaries during grain growth [47], and the transition to abnormal grain growth [48]. Using Kingery and Francois' model relating pore curvature, dihedral angle, and number of grains surrounding a pore [49], Lange suggested that grain growth may be necessary to lower the pore coordination for pore shrinkage to be possible thermodynamically. While that concept is valid, calculations by Zhao and Harmer [29] and by Evans and Hsueh [50] indicate that the sintering kinetics for large pores are so slow that even when a large pore can shrink thermodynamically, grain growth is ineffective in promoting densification. Other dihedral angle effects on sintering are discussed in more detail elsewhere [51]. These new models incorporating dihedral angle effects are especially timely since measurements of the free surface-grain boundary dihedral angle demonstrate that the average dihedral angle in undoped Al_2O_3, MgO-doped Al_2O_3, and MgO polycrystals is in the range 106° - 117°, not 150° as previously measured [52-53] Measurements of dihedral angle by Ikegami et al. show the same trends but the average values are slightly higher [54].

Our old workhorse, Al_2O_3, has been used to examine the effects of pore size, dopants, and liquid phases on pore-boundary separation and the orientation dependence of grain growth. Model experiments of a single crystal sapphire growing into a fine-grained matrix have been performed using sapphire crystals with c-axis, a-axis, and 60° off c-axis cuts in contact with dense polycrystalline samples [55-59]. The matrices examined are: with and without arrays of pores at the single crystal-matrix interface, with [55,59] and without [56-58] a silicate liquid, and with and without MgO. An important feature of some of these studies is the controlled etching of pore sizes and shapes in the sapphire surface using photolithographic techniques in order to measure accurately the effect of pore size and pore spacing on the conditions for pore-boundary separation [56-58]. The results include:

1. MgO doping to Al_2O_3 without a liquid appeared to increase D_s, thereby increasing pore mobility.
2. The pore spacing is a critical variable for breakaway with pores in undoped Al_2O_3 remaining attached to the growing sapphire (c-axis cut) for pore spacings less than 6 μm and for times up to 20 hrs.
3. In MgO-doped Al_2O_3, pore arrays moved with the growing interface at a faster velocity but pore-boundary separation occurred at an annealing time of 10 hrs, independent of pore spacing.
4. Without a liquid, c-axis sapphire grew into an MgO-matrix faster than into undoped Al_2O_3.
5. In the presence of a silicate liquid, c-axis sapphire grew more slowly than a-axis sapphire, with the difference in velocity between c-axis and a-axis increasing as the Ca to Si ratio increased. The authors suggested that this difference in velocity results from the process being interface controlled.

The latter notion of interface control conforms to Burke's suggestion that, since MgO is not found at grain boundaries in Al_2O_3 (but is so effective at suppressing breakaway grain growth) that the model for crystal growth on well-developed facets with steps and ledges and kinetics should be adopted for Al_2O_3 [60]. Thus, a very small amount of MgO at the steps/kinks could "poison" growth at special growth sites. However, the observation that MgO-additions increase the boundary mobility of c-axis sapphire without liquid or pores, summarized above, is in direct conflict with this notion. More work is required to address some of these discrepancies.

The effect of second phase particle pinning of grain boundaries during grain growth has been studied through experiments on Al_2O_3 with FeO particles [61], ZrO_2 with Al_2O_3 particles [62], and Y_2O_3 doped with La [63]. FeO and La-yttria phases appear to inhibit grain growth while Al_2O_3 particles in ZrO_2 have little effect on grain growth. A new evaluation of the Zener criteria for boundaries pinned by pores for various microstructures and theoretical formulations establishes the ranges over which different pinning equations are applicable [64].

It has become widely accepted that many ceramics earlier thought to be single phase bodies in fact contain liquids at the sintering temperature due to impurities in the powder or introduced during powder processing and sintering [65-71]. These liquids degrade the creep resistance at high temperatures and the uncontrolled amounts of liquid lead to wide variations in measured properties. For example, abnormal grain growth in undoped Al_2O_3[68] and Fe_2O_3 [65] was induced by unintentional silicate-based liquid phases. This has led to increased attention to the initial chemical powder composition and to control of contamination by clean-room processing [72-77]. In addition, organic inclusions introduced during processing may leave no chemical residue but may cause damage to the matrix during sintering or themselves comprise flaws after sintering that seriously degrade the mechanical properties at low temperatures.

Other recent theoretical studies have examined the kinetics and thermodynamics of particle rotation during sintering [78] and the contiguity of sintered structures [79]. Of particular interest from an engineering point of view are two studies on the non-destructive characterization of the progress of sintering using elastic property measurements [80] and SANS [81].

COMPOSITES

There is a natural link between sintering of single phase powders and composite sintering: any variability in the shape, particle size distribution, and degree of agglomeration in a powder may lead to inhomogeneous sintering resulting from heterogeneity stresses. Theoretical analyses have determined that inclusions with different densities than the matrix will retard the densification of the matrix by the creation of a hydrostatic tensile stress in the matrix of the order of 0.5 to 2 MPa [15,82-89]. For particulate composites, a rough estimate of the magnitude of the tensile stress is the volume fraction of the particles multiplied by the sintering stress, Σ. The magnitude of the heterogeneity stress was verified experimentally by Ostertag from the bending during sintering of Al_2O_3 compacts reinforced in an asymmetrical pattern with SiC fibers [90,91]. As determined both theoretically and experimentally, increases in the fraction of heterogeneities in the matrix or in the difference in initial density between heterogeneity and matrix lead to a increasing suppression of matrix sintering rate and lowering of the final density.

The number of different composite systems and experimental variables examined is impressive. Among the many studies of systems where the matrix and the dense inclusions are the same material are: dense MgO cylinders in MgO powder [92]; alumina agglomerates in alumina [93]; yttria agglomerates of various strengths in yttria powder [94]. Sintering of dissimilar materials has been examined in systems as diverse as SiC fibers, whiskers, or particulate in Al_2O_3 [95], Al_2O_3 agglomerates in TiO_2 [96], TiC-reinforced Al_2O_3 [97], BN-reinforced SiC [98], mullite produced by sol-gel processing reinforced with SiC whiskers [99-102], ZrO_2 particles in Al_2O_3 [103], B_4C-TiB_2 composites [104], and SiC-reinforced glass [105].

Most theoretical work has focused on identifying stress generation in the matrix resulting from the difference in densification between the matrix and the inclusion/agglomerate. The stress field generated by differential sintering around a spherical dense inclusion is composed of a tensile hoop stress and a radial compressive stress, with a tensile mean stress [82]. The magnitude of the stress depends on the relative rates of creep and matrix densification, and microstructural variables, such as the volume fraction and size of the inclusions. The creep rate is typically described in terms of a matrix viscosity which changes during densification [106- 113]. If the creep rate and densification rates are controlled by different mechanisms with different activation energies, the effect of dense inclusions may be changed by changing temperature. On the other hand, if same mechanism controls both creep and densification, then changes in temperature will produce no benefit.

In experiments on viscous matrices with dense inclusions, there appear to be three regimes of behavior as a function of inclusion volume fraction. At volume fractions ≤ 0.1, the densification rates of the composites can be described by a simple rule-of-mixtures. Only over a narrow range of inclusion volume fractions (between 0.1 and 0.12) are the results in agreement with the Scherer's theory for viscous sintering with rigid inclusions. However, for higher volume fractions, the densification rate deviates significantly from either of these models. In experiments on crystalline matrices with dense inclusions, the measured densification rates are significantly lower than predicted by theory over all ranges of inclusion volume fraction [86].

The explanation for this discrepancy between theory and experiment remains controversial. Scherer has argued that the stresses are of the same order as the sintering stress and cannot, therefore, explain the magnitude of the effect in polycrystals containing many heterogeneities/inclusions [111]. In addition, application of a hydrostatic pressure only slightly greater than the tensile hydrostatic stress is predicted to eliminate the effect of the inhomogeneities. However, retardation of matrix densification is observed for small volume fractions and for particles as small as the matrix particle size [86]. Bordia and Raj found that the application of a low quasi-hydrostatic pressure <10MPa would suppress damage formation in the matrix but does not restore the matrix densification rate to the rate without heterogeneities [114].

One plausible argument is that heterogeneities are imperfectly distributed so that heterogeneities form interconnected structures over short distances in the matrix. The percolation limit for a given particle shape and size distribution gives an estimate of the particle volume fraction when particles form a completely interconnected structure that cannot densify. For single-sized, spherical particles, the percolation limit is at a volume fraction of 0.16. At volume fractions smaller than the percolation limit, inhomogeneities will sinter together and, thereby, limit the density of matrix in the vicinity of the sintered heterogeneities. An additional explanation is that the powder packing density near a heterogeneity may be lower than in the matrix away from the inclusion and will, therefore, lead to a lower limiting density [45,84]. The magnitudes of these two effects must be calculated to assess their contributions to the observed suppression of matrix densification rate.

An alternate explanation proposed by Mataga is that the sintering in heterogeneous powder is not properly described by a linear, isotropic sintering model [115]. Mataga suggested that a non-linear material model may be required as a result of non-linear stress dependence on sintering rate, a creep threshold, or non-linear creep. He noted, however, that significant deviations from linear behavior are necessary to explain the experimental results. (Additional factors may be a change in the diffusion path due to the sintering stress [111] or a change in the coarsening/sintering trajectory in the presence of dense inclusions.)

In light of these theoretical and experimental results, Rahaman and Jeng have suggested practical guidelines for minimizing damage formation in composite systems [102]:

1. The packing in the matrix should be maximized.
2. The inclusions/agglomerates should be distributed *uniformly* not *randomly*, in the matrix to minimize inclusion/agglomerate interactions.
3. The optimum sinterability conditions for the unreinforced matrix should be used for sintering the reinforced composite.
4. If the powder is amorphous, the composite should be densified before crystallization occurs.

Of these four guidelines, (3) is the most provocative. What really is the proper sintering schedule for a composite as compared with the matrix without the reinforcing material? What is the effect of coarsening - to strengthen the necks before densification or to narrow the particle size distribution before densification and grain growth begin ? For materials that sinter easily under a variety of conditions, such as the classic model material Al_2O_3, the sintering heat treatment for composites may be very different from that which produces the highest final density of the matrix alone.

An alternative approach to preventing damage formation in composites is to allow the matrix to sinter to almost full density before it comes into contact with the fiber by creating a void space of controlled thickness around the inclusion. This void space can be produced by coating the dense inclusion with an organic which burns off before sintering. This approach also has the advantage that the bonding between the fiber and the matrix can be controlled by the initial gap thickness [106].

It should be noted that in the previous section, the systems chosen to examine stress generation in the matrix exhibit little or no intermediate phase formation between the matrix and the reinforcing materials. In many engineering situations the only possible materials for a specific application may react and it is the ceramic engineer's task to minimize the extent of reaction by careful, innovative processing.

An additional factor influencing the sinterability and final properties of composites is thermal expansion anisotropy when the same material is used for matrix and reinforcement and thermal expansion mismatch when the phases of the matrix and reinforcement are different. During heating of composites, neck formation begins at the same time that stresses develop. The stress generation during heating will then depend on the ratio of the creep rate required to relieve thermal expansion mismatch stresses and heterogeneity stresses to the densification rate.

LIQUID PHASE SINTERING

The progress in theory and experiment for solid state sintering summarized above has been matched by similar accomplishments in the field of liquid phase sintering. Before citing specific research topics, we would like to acknowledge the completion of a major work in liquid phase sintering: the book *Liquid Phase Sintering* by Randall M. German [116]. This textbook is an excellent general reference which is, as Prof. German states, truly materials-independent and appropriate for engineers and scientists with diverse technical backgrounds. We congratulate Prof. German on his accomplishment and recognize his significant contribution to the field.

The past five years have brought a better understanding of the processes occurring in the neck region where two solid particles are in contact with liquid, in assemblages of particles filled with liquid, and in the transient state when the liquid first becomes molten. The calculation of the equilibrium configuration of particles and liquid and the forces generated by a liquid meniscus was demonstrated by Heady and Cahn for liquid phase sintering of spherical and jagged particles [117,118]. The calculation is based on an energy minimization subject to certain conditions, such as volume conservation. Recent calculations by Park, Cho, and Yoon have used this approach to model the filling of isolated pores by liquid during liquid phase sintering [119]. Grains were assumed to maintain their equilibrium shape determined by the balance between the tendency of grains to become spherical ("sphering force") and the negative capillary pressure due to the liquid menisci. The filling of pores and contact flattening were found to depend on liquid volume fraction and grain size, thereby leading to greater pore filling as the grain size increases. This gradual pore filling as grain growth/coarsening proceeds explains the experimentally observed agglomerate formation during the early stages of liquid phase sintering. The thermodynamic criteria for sequential pore filling in different 2-D particle geometries were also examined by Shaw [120].

From Park, Cho, and Yoon, an additional result of special importance is that particles completely surrounded by liquid but having a vacuum-filled pore in the center of the liquid is an unstable equilibrium. An infinitesimal perturbation leads to filling of the spherical void. In contrast, if the pore is filled with an insoluble gas, the pore will reach a stable equilibrium size. This is contrary to the liquid phase sintering model previously derived by Kingery [121]. The observation of pores completely surrounded by liquid means that an entrapped gas is limiting the final density, and that removal of the gas by alternate processing techniques will allow the structure to achieve a higher density.

A major advancement in liquid phase sintering, as well as in high temperature materials properties, is the recognition that surface forces exist between two solid surfaces forming the neck region between two sintering particles. The surface forces have many sources, for example, electrostatic interactions, van der Waals attraction, or structural (steric) forces, and can lead to an equilibrium thickness of liquid separating the two solids. The measurement of surface forces between ceramic surfaces at room temperature is an active research area, made possible by the development of a technique for measurement of surface forces. For liquid phase sintering, the nature of these interactions will affect liquid redistribution as the volume fraction and/or grain size of the particle change and will, in many cases, determine the mechanical properties at high temperature.

Clarke has examined the equilibrium thickness of a thin intergranular where structure within the liquid determines the surface forces [122]. In a complementary experimental study by Greil and Weiss, the equilibrium thickness of thin liquid silicate layers in β-SiAlON was found to be constant for increases in the liquid volume fraction

from 2% to 10% [123]. In addition, structure in a thin intergranular liquid phase was detected in an anorthite-based liquid in Al_2O_3 [124].

In most of the theoretical treatments described above, equilibrium configurations are the endpoints of the calculations. In liquid phase sintering experiments, the creation of equilibrium configurations is preceded by solid state sintering, disintegration of the sintered skeleton as melting occurs, mixed fluid and particle flow, particle dilatation, rearrangement, and finally to disintegration of the individual particles [125]. Grains will grow by grain growth/Ostwald ripening and coalescence processes. Grain shape may also change due to facetting transitions and/or growth rate anisotropy in the presence of a liquid phase. The identification of each stage is important in relating changes in processing variables to changes in microstructure. Selected examples of these processes are presented below.

As with solid state sintering, the magnitude of the sintering stress that provides the driving force for liquid phase sintering is an important parameter in determining sinterability. The magnitude of the sintering stress has been calculated by De Jonghe and coworkers for liquid phase systems, as well as for solid-state systems, from the ratio of the densification rate at no load to the creep rate with an applied uniaxial load. In the $MgO-Bi_2O_3$ system, De Jonghe and Srikanth found that the sintering stress was a constant throughout densification [126]. The calculated stress was low - 0.08 MPa - corresponding to a meniscus radius of curvature of about 6 μm, a value much larger than the MgO particle size. The low value of the sintering stress was attributed to formation of agglomerates by the sequential pore filling as suggested by Shaw and by Park, Cho, and Yoon.

In the final stages of sintering, grain growth/coarsening/Ostwald ripening, discontinuous grain growth, and densification occur simultaneously. Three recent studies illustrate the effect of the liquid phase on discontinuous grain growth and pore coalescence. In a study of grain growth in TiO_2-rich $BaTiO_3$, Hennings, Janssen, and Reynen found that the grain size of abnormally growing grains decreases with increasing additions of TiO_2-enriched seed grains[127]. Also, the grain size increases with increasing TiO_2 content to maximum at 2 mol% excess TiO_2. As noted above, studies of sintering and grain growth in undoped Al_2O_3 and Fe_2O_3 revealed that discontinuous grain growth can originate from regions containing a calcium-alumino-silicate second phase [65,68]. These results suggest that discontinuous grain growth is accelerated by a non-uniform distribution of liquid phase. A study of normal grain growth and pore coalescence in the $MgO-CaMgSiO_4$ system demonstrated that the coalescence of gas-filled pores is controlled by the rate of grain growth/Ostwald ripening of the solid grains [128].

An important feature of many liquid phase sintering systems is the facetting and shape change of the solid particles when they come into contact with the liquid. For example, pronounced facetting has been observed in anorthite- Al_2O_3 [59, 129, 130] and in β'-sialons [131]. The same growth phenomena identified for crystal growth in systems with facetted liquid-solid interfaces are expected to operate in these liquid phase sintering systems [132]. Among these phenomena are: (1) growth on a facet can be limited by the nucleation of a stable ledge, also known as "2-D nucleation limited growth"; (2) growth and dissolution shapes are not expected to be the same; (3) screw dislocations and special twin boundaries can serve as nucleation sites for growth in slow growing directions.

EFFECTS OF APPLIED PRESSURE: HOT PRESSING, HIP'ING, AND SINTER-FORGING

The industrial use of applied pressure to densify a wide variety of difficult-to-sinter materials has increased over the last several years. This has coincided with a increase in modelling of the effects of applied pressure on sintering [133-140]. These models fall into two different types. There are models based on the visco-elastic response of the porous compact to the applied stress. The constitutive equations for the entire body over the whole range of deformation are specified and then the response is numerically calculated [133-137]. An alternative approach is to divide the densification up into different regimes, as shown in hot pressing or hot isostatic pressing (HIP'ing) maps [138-140]. Within a regime a single mechanism will dominate, and the dividing lines between regimes are where the rates of densification are equal. The conditions required to obtain a desired microstructure can be estimated from trajectories on maps. The results of both techniques are consistent and provide insight into the densification process and the observed shape changes during hot isostatic pressing (HIP'ing). For example, a densification front moves inward during HIP'ing as densification proceeds. The main reason for the development of this densification front is a nonuniform temperature distribution during heating. The hotter material near the outer edge will densify faster. The heat conduction through the dense outer region can further change the temperature distribution. Also the dense outer region can support some of the load and thus reduce the pressure on the porous interior. These effects can lead to very anisotropic shape changes during HIP'ing. To study the densification in a systematic manner, model experiments have been preformed by Kaysser and co- workers [125]. The deformation of a single, polycrystalline particle under an applied uniaxial load was studied. In this technique they were able to measure changes in the geometry during densification.

The effect of nonuniform stresses during sintering is not clear. The addition of a shear stress, or uniaxial stress to a compact during sintering has yielded results which suggest that the creep rate may be control the rate of densification. The applied stress increases the creep rate significantly, so that it is no longer rate-controlling [141,142]. This effect is of importance only when creep is the rate limiting process which, for ceramics, occurs in a very limited number of systems. A more pronounced effect of shear stresses is an enhancement of the rearrangement during sintering. Large processing defects can be broken down during shear deformation, but would be stable during cold isostatic pressing [143]. Such large defects can also be removed during HIP'ing if the material is in a regime where extensive plastic deformation can occur, either by diffusion of dislocation motion [144-146].

A wide variety of materials which are difficult to densify at atmospheric pressure have been successfully sintered with the application of moderate to high pressures [147-150]. HIP'ing has been used to great advantage for removing defects in sintered

metallic materials. It has been noted however, that near surface defects may increase in severity during post- sintering HIP'ing [151]. The development of HIP maps, and the underlying data base necessary for their use, has allowed the conditions to achieve high density to be estimated in advance of the experiment [152,153].

While experiments have shown the tremendous benefits of applied pressure on sintering, there are many limitations. The most severe is the bloating, or desintering problem associated with many HIP'ed or hot pressed materials. The bloating is due to two main effects. Oxidation of impurities which were incorporated during pressing produces a internal gas pressure which causes pore growth. This can be avoided by restricting use to inert environments, but this limits the usefulness of the materials. Another source of bloating is due to pores which remain in the material after pressing. These pores are due either to trapped residual gas or to thermodynamic stability of the pores as a result of the surface energy to boundary energy ratio [52,121]. While the applied pressure will reduce the size of such defects as compared to pressureless sintering, when the pressure is removed the defects are not in thermodynamic equilibrium and at elevated temperatures, they will grow until they are in equilibrium. This again limits the use temperature of a HIP'ed material. This effect implies that the minimum pressure possible should be used, and that the major benefit due to pressure will be an increase in the densification of large defects.

CASE STUDIES

Zirconia

Zirconia, one of the toughest ceramic materials, has mechanical properties which are dependent both on microstructure and chemistry. Additives are required to stabilize the tetragonal phase and give high toughness. Also, the grain size must be kept small to retain the metastable tetragonal phase at room temperature. For a detailed review of recent work in zirconia see Somiya et.al.[154]. Zirconia is a material which sinters well. Densities above 99% are achieved under a wide range of processing conditions and dopant concentrations. [1] Two important factors in determining the time and temperature required for densification are the starting powder particle size and the degree of powder agglomeration, as summarized below.

Ultrafine zirconia powders (10-20nm) forming 1-10 μm agglomerates can be produced by chemical precipitation [155,156] and these powders sinter to full density at 1400°C in 2 hrs. At the initial stage of sintering, density decreases with time [156] resulting from differential densification in the agglomerated powder. Density increases when grain growth begins. This coarsening at the initial stage of sintering is not disastrous in this system due to the extremely small grain size, even after grain growth. Rhodes [20] demonstrated that deagglomeration of ZrO_2 can reduce the time and temperature required to sinter a commercial power from 4 hours at 1500°C to 1 hour at 1100°C. An additional factor limiting final density is that gas evolution from residual organics used in washing the powders can cause bloating/desintering at higher

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[1] One difficulty in interpreting the density data is the fact that the changes in the fraction of the different phases lead to changes in density without any change in porosity.

temperatures. Powders produced by electro-refining and grinding [157] have similar ultimate densities and properties as compared with chemically produced powders, but with somewhat reduced sintering rates. This allows a greater degree of control over the dimensional stability of the material.

Additions of Ce, Y, Ca, Mg, or Ti to stabilize the tetragonal phase do not reduce the sinterability of the powders [158-163] and, if Si is present as an impurity, may increase the densification by the formation of a silicate- based liquid phase. Also, the addition of 40% ZrO_2 to ZrC allowed the densification of the composite at 2000°C [164] if the green density was >55%.

Sinter forging of zirconia powders [165] determined that densification can occur by plastic flow at pressures and temperatures of 1400°C. Through hot forging of dense materials [146] it was found that fine- grained zirconia could be deformed but large grained zirconia could not. Diffusion control was thought to be limiting, probably due to the limited number of independent active slip systems. Thus for plasticity, diffusional accommodation is necessary and is only possible in fine-grained materials at the strain rates used. Sintering followed by HIP'ing has been used to remove large pores (30-40μm) and achieve full density at 1200°C [144], most likely due to plastic flow in addition to increased driving force for densification. It is necessary to achieve high density (>96%) before HIP'ing to prevent open pores from limiting the final density.

Dielectric ceramics

For production of dielectric ceramics by sintering, the critical issue is control of the microstructure. While many additives have been introduced to improve the electrical properties, recent work has focused on additives added to improve the sinterability and retard grain growth. Typically the additives promote the formation of a liquid phase and thus allow sintering to occur at low temperature [166-169], but may also work by reducing the vapor transport [170]. In the Pb-Mg-Nb-O system, avoidance of the pyrochlore phase is important, and the presence of liquid phases [171-173] controls not only the microstructure but also the phase content of the samples. With the formation of a liquid phase, the heating schedule becomes important: fast heating rates may lead to an inhomogeneous distribution of liquid phase [174].

Many dielectric materials have high vapor pressures at the sintering temperatures. This leads to weight loss and compositional changes during sintering. Some additives reduce the volatilization [175], but the problem is usually solved by adding an excess of the volatile components. Fast firing can reduce the problem as the material spends less time at temperature, and, therefore, has a finer grain size [176] than conventionally sintered PZT materials.

Different sintering atmospheres may also change the sintering behavior [177]. This can be for a variety of reasons. Often the coarsening rates can be reduced in specific atmospheres. In certain cases the phase relations may change as the atmosphere changes. Another possibility is that the defect concentrations change with atmosphere and thus the diffusion coefficients also change. All these effects are very system specific and need to be examined for each new system. A knowledge of how specific effects influence sintering can be used to estimate the behavior from other measurements.

While high density is important, removal of all porosity is not as important to electronic properties as having a small grain size. The presence of a liquid phase can promote exaggerated grain growth in $BaTiO_3$ [69], as well as in alumina, discussed above, and in $Ba_2YCu_3O_{6+x}$. In ferroelectrics large grains are generally not desired due to domain migration within the grains. Also, for non-cubic materials, microcracking due to thermal expansion anisotropy is reduced at small grain sizes [178]. Some of this effect can be eliminated by texture development produced by hot pressing [179]. While hot pressing or hot forging lead to better microstructures, use of pressure sintering is precluded for most electronic applications.

ZnO Varistors

In ZnO varistors, the presence of a grain boundary phase controls the properties. The distribution of the intergranular phase is influenced by the composition, sintering temperature and atmosphere [180]. In pure ZnO, various additives have been shown to affect the relative rates of coarsening verses densification [181]. As discussed above, this may be advantageous in promoting neck growth to prevent cracking due to differential densification.

Ceramic superconductors

While there have been many studies of ceramic superconductors, few have systematically examined their sintering behavior. It is now clear that the formation of liquid phases at high temperature often controls the microstructure evolution [182]. More definitive sintering studies are difficult to perform because the liquidus is strongly dependent on composition and atmosphere [183-185]. Although a variety of techniques have been used to improve the properties of ceramic superconductors [186- 190] with differing degrees of success, microstructural control in these systems will require careful control of all the processing parameters.

Ferrites

In most applications for ferrites, the important properties are the magnetic properties which are limited by the microstructure. Small grain sizes and pore-free materials are generally desired. These features are controlled most often by controlling the atmosphere during sintering to both suppress volatilization [191] and to affect diffusion coefficients through changes in the defect chemistry [192-194]. For a review of the current state of processing of ferrites, see Wang [195]. The use of an acicular powder leads to a smaller grain-sized dense Ni-Zn ferrite than the use of conventional powders [196]. The sintering of ultrafine powders also resulted in Ni-Zn ferrite and Co ferrite [197] with submicron-sized grains. Reactive sintering of Zn ferrite was not at all useful [198] but rather produced microstructures worse that conventionally processed powders. The use of a low temperature melting phase (Bi_2O_3) was shown to yield optimum electrical and magnetic properties for the case of 1% additions to Li-Ti ferrites [199]. The properties declined for larger Bi_2O_3 additions.

Silicon Carbide and Silicon Nitride

The development of techniques for sintering of SiC and Si_3N_4 has been reviewed by Greskovich and Prochazka [200]. They give the sintering conditions for selected compositions, with an emphasis on describing the conditions which control densification. In both these technologically important materials, coarsening mechanisms compete with densification mechanisms. Achievement of high density requires suppression of the coarsening processes. A wide range of conditions for a variety of compositions have been examined by many other authors [201-217]. Effects of additives, oxygen content, homogeneity, precursor type and size, temperature and sintering atmosphere have been considered. While these materials cannot be sintered without additives at atmospheric pressure, materials with excellent properties can be obtained by careful selection of the initial powders, the additives and the sintering conditions. Again, the optimum sintering conditions depend on the desired properties of the sintered body [217,218].

Special techniques

In addition to pressureless sintering, hot pressing and HIP'ing, there are several other techniques which have been used to produce dense materials. Very high pressures have been used for sintering several difficult-to-densify materials. Self combustion sintering with an applied pressure of 3GPa has been used to densify TiC [219], diamond has been densified at 7.7GPa and 2000°C [220], and BN has been densified using shock compaction [221] with pressures estimated to be over 33GPa. These pressures are beyond the pressures normally obtained in commercial systems and will only be applicable to special systems. Microwave and RF plasma sintering has been successful for sintering a variety of materials, such as Al_2O_3 [222-224], Al_2O_3-ZrO_2 composites [225], ZrO_2 [226] and ferrites [227] in short times. Shock activation of powders before sintering has been shown to have beneficial effects on the densification of AlN [228] and Al_2O_3 [229]. Vapor phase transport can also contribute to enhanced densification when materials are sintered in a temperature gradient [230-232]. There is a net migration of the sample down the temperature gradient and material is deposited in the neck surface in the cold regions after leaving the particle surfaces in the hot region. Thermal cycling effects on densification are though to be due to temperature gradients in the sample while it is equilibrating [233].

Reactive sintering

The formation of composites by direct reaction of liquid metals [234,235] is a unique method for producing large components. Composites of ZrC and reinforcing ZrB_2 platelets have been produced with porosities less than 1% by reaction between liquid Zr metal and B_4C. A wide variety of ceramic materials have been sintered under conditions where a reaction is also taking place. In many cases the reaction aids the densification process, but in some cases the reaction is not desired and the conditions need to be manipulated to reduce the amount of reaction. B_4C- TiB_2 composites sintered with Fe additions resulted in dense (>97%), fine grained material [236]. Fe formed a liquid phase which aided densification and excess TiB_2 inhibited grain growth. In the Al_2O_3-TiC system, the reaction produces CO_2 which reduces densification [97]. However, rapid heating minimized this effect by reducing the time spent in a temperature range where the reaction occurred, and dense materials were obtained. In reactive sintering of ZrC-ZrO_2 composites, the amount of excess ZrO_2 was found to be important in determining the density of the sintered material [237]. In the case of Cr_2O_3 with TiO_2 additions, the densification depended on the atmosphere, with little densification in air and increasing as Po_2 decreased [238]. It was felt that this was due to a change in the defect chemistry rather then suppression of Cr volatilization. The effect of H_2O on the sintering of MgO is not well understood [239]. Closed pores were observed to shrink while the open pores coarsened, indicating that H_2O may activate surface or vapor transport. In the $MgAl_2O_4$-SiO_2 system, the sintering mechanism changed with temperature [240]. At low temperatures, viscous flow of SiO_2 dominated until the formation of crystobalite and then diffusion controlled the densification. At higher temperatures the formation of cordierite caused an expansion of the sample, and the grains were spinel cores surrounded by cordierite. Dense Al_2TiO_5 can be formed by reactive sintering of Al_2O_3 and TiO_2 [96,241], but careful control of the sintering temperature was required.

Very porous mullite gels (44% dense) can be sintered to 97% density but the resulting structure consists of elongated grains of $3Al_2O_3$- 2SiO2 and fine grained Al_2O_3-$2SiO_2$ [101]. The structure of the gel may have an effect on the final microstructure produced [242]. Seeding of the gels to provide nucleation sites for the reaction proved effective in getting dense materials with a uniform grain size [243,244]. Seeding has also been effectively used for Al_2O_3 [244] and Al_2O_3-ZrO_2 composites [99].

SUMMARY

Significant advances in modelling of solid-state and liquid-phase sintering and grain growth have been made. The major driving force behind these advances has been the realization that fundamental thermodynamic concepts could be extended beyond what had been done for the two-sphere model. In particular, models of pore filling in multiparticle groups for liquid phase sintering and of the stability of a row of sintering spheres have provided new insight into the processes governing sintering. Improved powder processing has lead to the creation of closer-to-ideal microstructures with which to test theoretical predictions of models that assume uniform microstructures.

A major shift in research has been made to sintering in the presence of intentional and unintentional non-densifying inclusions. Modelling and experiment have examined the effects on the densification of a finer grained matrix by large particles (or agglomerates) of the matrix material and of dense particles, whiskers, or fibers of a different phase intended for reinforcement. Even for small volume fractions of inclusions, the matrix sinterability is seriously impeded. Strength-controlling defects can form in the matrix as a result of sintering in the presence of dense inclusions. Because of the severity of matrix damage during sintering, the analysis of damage formation during sintering and, in particular, the factors which inhibit crack formation are topics of wide interest. So far the theoretical estimates of the stresses produced by these heterogeneities do not seem to agree with experiment. However, there is a general recognition that application of constitutive laws is required for properly describing constrained sintering in single- and two-phase bodies.

For many composite systems, hot pressing may be required to produce high density materials. Unfortunately, applied pressure is not a panacea for composite systems: damage to high aspect ratio whiskers or fibers during pressure-sintering may degrade the mechanical properties of the composite and the applied pressure does not restore the sinterability to the level without inclusions. However, hot pressing and HIP'ing are viable techniques for producing ceramic parts. For example, a commercial cutting tool has been manufactured by hot pressing SiC whiskers in an Al_2O_3 matrix.

Of the recent research on hot pressing and HIP'ing, the generation of hot pressing and HIP'ing mechanism maps has the greatest impact on the use of pressure-sintering techniques in manufacturing. Although an extensive data base is required for the generating these maps, the data base is smaller than for the corresponding sintering maps/diagrams, since the number of mechanisms which dominate tends to be small when pressures are applied. These maps can also be used on-line in research and development to follow the densification process and to determine changes in processing conditions in real time.

When pressureless sintering, hot-pressing, and HIP'ing are not effective, or a special system presents unique opportunities, different techniques for processing should be used. The use of reactive sintering, shock activation, microwave sintering, self-combustion sintering and plasma sintering is of advantage only in the systems which allow the processing to exploit the special characteristics of these methods.

The case studies presented demonstrate the use of the fundamentals outlined in the beginning of the paper. Knowledge of the relationships between the observed microstructure and the processes which give rise to such microstructures has allowed the processing to be manipulated to achieve a specific microstructure. Without this understanding of the fundamentals of sintering, improvement of the properties of a specific material must occur by trial and error and in an incremental manner. Sintering has now progressed to the point where material development can be done more systematically to optimize properties.

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