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PHOTOLITHOGRAPHY: A NEW TOOL FOR CERAMIC SCIENCE

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ABSTRACT

A recently developed technique combining photolithography, ion beam milling, and hot pressing allows the production of submicron-scale controlled-geometry, controlled-crystallography pore structures in controlled misorientation bicrystals, single crystal-polycrystal ensembles, and polycrystal-polycrystal ensembles. Such microdesigned interfacial structures provide a new tool for studying fundamental aspects of microstructural evolution. Current applications of the technique, opportunities for future research, and future extensions and refinements of the technique are described.

INTRODUCTION

The microstructural changes that occur during sintering and high temperature use of ceramics are generally complex. In real systems, the path of microstructural evolution reflects the outcome of a competition between many concurrent processes. For sintering, particle size, particle size distribution, and particle packing play a major role in determining the pore size, pore size distribution, and pore spacing in the green compact. These characteristics determine the thermodynamic driving forces for both coarsening and densification processes. The coupling between these topologically dictated driving forces and the transport coefficients for grain boundary, lattice, and surface transport, or interfacial reaction rate constants, or both, will ultimately determine the relative fluxes associated with competing mass source-sink pairs, and thus will determine the dominant transport mechanism.

To make theoretical modelling of microstructural evolution tractable, it is often necessary to simplify the system. For example, a single process is assumed to dominate, the properties of the system are assumed to be isotropic, and in many instances, the geometry is simplified. The systems that are dealt with in practice are much more complex. As indicated, multiple processes interact. Moreover, the materials are rarely isotropic, and the geometry of the microstructural features of interest is rarely as simple as it is assumed to be.

When significant disparities exist between theoretical predictions and experimental observations, the origin of the disparity can be difficult to pinpoint. Is there a fundamental error in the modelling, or is the disparity the result of geometrical differences and competing processes that are not accounted for? Studies on real materials can be inadequate to test theoretical models critically because the degree of control over the relevant microstructural geometry is inadequate. Thus, an experimental method that provides control over the pore, flaw, or second phase geometry has the potential to further our understanding of microstructural evolution during sintering and high temperature use.

Our recent work has focussed on developing a new family of model experiments that utilize photolithography to produce geometries that more closely resemble those that have been theoretically modelled. Our goals are: 1) to design model experiments that permit the systematic study of particular processes under well-defined conditions, 2) to utilize these experiments to verify theory, or to identify important deficiencies, 3) to provide information that can be useful to the materials processing community, and 4) to help bridge the gap between theory and practice.

The following paper provides a brief overview of the sample preparation procedure, and then summarizes the results of new model experiments examining high temperature crack healing, pore-boundary interactions during sintering, and pore coarsening and pore elimination. Potential extensions and refinements of the method are cited, and some future directions are suggested.

SAMPLE PREPARATION

Lithography has furnished a new tool for studying a number of processes that occur during fabrication or during use at high temperature. The combination of photolithographic methods, ion beam etching, and hot pressing provides the ability to define and introduce surface features with a controlled geometry and location, and to subsequently transform these surface features into internal features. A brief overview of the procedure follows. A more detailed description of the experimental methods has been published elsewhere [1,2].

The basic processing steps are illustrated schematically in Figure 1. A single crystal wafer or a polycrystalline specimen is coated with a uniformly thick photoresist layer. For the studies that will be described, sapphire single crystal wafers were used, however, the technique has much broader applicability. Using a typical range of processing conditions, the thickness of the photoresist can be varied from 1.3 to 2.6 μm . The photoresist is then selectively exposed using uv radiation. The shape of the exposed features reflects the mask pattern. The geometry of features on the mask is defined using pattern generation software. For positive photoresists, exposure increases the solubility of the photoresist, and allows its selective removal, thus exposing the substrate. Ion beam etching is used to etch the exposed ceramic surface, and thereby transfer the pattern to the ceramic substrate.

For sapphire and alumina, an etched and an unetched sample are bonded by hot pressing (1370°C, 60 min, 15 MPa, $2.6 \cdot 10^{-3}$ Pa), thereby transferring the surface pattern to an internal interface. A wide variety of ensembles can be produced to address a broad range of experimental interests. Specimens prepared by bonding an etched single crystal to a polished single crystal of identical orientation provide a means of simulating defects in single crystals, intragranular flaws. Controlled misorientation bicrystals can be used to study intergranular defects. Single crystal-polycrystal ensembles and polycrystal-polycrystal ensembles provide an opportunity to examine the effects of a wider range of misorientation, and to simulate and study defects in typical polycrystalline materials.

Examples of the types of surface structures that can be generated are provided in Figure 2. Features with minimum dimensions of order one to two microns can be produced using conventional lithographic procedures, and can be repeated up to 10^6 times on a single substrate. The feature depth is typically a few tenths of a micron, yielding minimum feature volumes that are $<1 \mu\text{m}^3$. The location of the features can be controlled to within $\pm 0.1 \mu\text{m}$. The specific experimental objectives influence the design of the surface structures. Those illustrated in Figure 2 have been utilized in studies of pore-boundary separation [3], pore coarsening and pore elimination [4], Rayleigh instabilities [5], and facetting [1,6].

Once surface features have been transferred to an internal interface, several methods can be used to study their evolution. When optically transparent materials such as sapphire are used, optical microscopy provides a convenient nondestructive mode of observation for all but the finest scale features. Using this method, a single sample can be given repeated anneals, and the time evolution of specific defects can be studied. When greater resolution is required, or when opaque material is used, fracture surfaces can be examined using optical or scanning electron microscopy. Finally, for the finest features, or when high spatial resolution is required, foils containing interfacial structures can be prepared, and examined using transmission or scanning transmission electron microscopy.

APPLICATION TO HIGH TEMPERATURE CRACK HEALING

The following section provides an overview of recent work on high temperature crack healing in sapphire. A more detailed account of this work is available in references 1 and 7. Work pertaining to the late stages of crack healing is described in greater detail in references 1 and 5.

Background:

Crack healing in oxides has been reviewed most recently by Gupta [8]. Most studies of crack healing have revealed that there are several geometrically distinct stages to the process. The initial stage is characterized by crack tip regression and blunting, sometimes accompanied by

facetting [9]. Discontinuous crack pinching can also initiate at cleavage steps or other irregularities along the crack front, or on the crack face [10]. Crack regression leads to the formation of a cylindrical ring around the crack periphery. This cylindrical rim is unstable to longitudinal perturbations. The second stage of crack healing is marked by the formation of "cylindrical" pore channels. Collapsed or healed portions of the crack perimeter propagate towards the crack interior, producing cylindrical ligaments whose axes are often perpendicular to the original crack edge. These cylindrical ligaments or channels are in turn subject to Rayleigh instabilities [11], leading to the formation of discrete spherical pores. Subsequent to the formation of isolated pores, pore elimination and pore coarsening can occur.

Several important variables affecting crack healing and strength recovery have been identified. The crack opening displacement can affect both the morphological characteristics [10], and the rate of healing [12]. The crack face crystallography can modify the healing behavior; facetting may aid or impede "cylinderization". Maruyama and Komatsu have demonstrated the sensitivity of the morphological evolution of cracks to grain boundary misorientation [13]. Crack face microstructure can also be important; Singh and Routbort have suggested that hillock formation accompanying grain boundary grooving can induce bridging of crack faces, and thereby contribute to strength recovery [14]. Finally, both crack healing studies [15] and measurements of surface diffusivities [16] in alumina have revealed a sensitivity of the healing and transport rates to impurities. Identification and quantification of the effects of particular impurities on crack healing could shed light on their effects on transport during sintering as well.

Although there are general behavioral trends, and some of the important variables have been identified, there are also pronounced system-to-system and even sample-to-sample variabilities in behavior that are not well understood. The difficulty in identifying the origin of these differences stems in part from a difficulty in controlling and isolating the effects of specific variables on the healing behavior. In view of the demonstrated importance of crack geometry, crack face crystallography, crack face microstructure, and sample chemistry/impurities on crack healing behavior, clarification and quantification of these effects seemed warranted. Our efforts focussed on applying lithographic methods to the study of crack healing, and subsequently assessing the importance of these variables on crack healing in sapphire.

Experimental Considerations:

For simulation of cracks, the features of interest are generally sufficiently large that the spatial resolution limit of the technique is not a concern. The accessible range of crack depth is a more important issue. During ion beam milling, both the ceramic and the photoresist are etched, and consequently, the relative etching rates of the substrate and photoresist determine the maximum feature depth. The etching rate of sapphire is approximately one-third that of the photoresist. Thus, using conventional processing procedures, crack depths approaching 0.9 μm are accessible.

The minimum feature depth is limited by the quality of the polished surface; lithographically introduced defects must have a depth exceeding that of the intrinsic polishing irregularities. Using as-received polished sapphire wafers, features as shallow as 40 nm can be transferred to an internal interface. With the use of atomically smooth substrates [17], this limit could be reduced further. Thus, it is possible to produce a wide range of cracklike defects with crack opening displacements similar to those in real materials.

Figure 3 illustrates a typical cracklike flaw introduced using lithography. The morphological evolution of geometrically similar cracks lying parallel to the basal (0001) and prismatic (11 $\bar{2}$ 0) planes was characterized to assess the effects of a difference in crack tip and crack face crystallography on the healing behavior. Up to 200 identical cracklike flaws were generated in each experiment. Most anneals were conducted at 1800°C (0.86 T_m) in a vacuum of $\approx 1.3 \cdot 10^{-3}$ Pa. In the work that will be illustrated, optical microscopy was used to study the morphological evolution of specific cracks within a particular interface.

Results and Discussion:

Figure 3 illustrates a typical cracklike flaw viewed in a cross section taken perpendicular to the crack plane. The flaw dimensions are 100 μm by 200 μm by 180 nm. The flaw was etched

into a basal plane, and after bonding, the sample was annealed for 10 min at 1800°C. The pronounced difference in curvature along the crack perimeter induces mass redistribution, which leads to recession of the crack perimeter, and the formation of an annular ring. Some faceting along the crack perimeter is evident, despite the high anneal temperature.

Figure 4 presents optical micrographs taken using transmitted light, illustrating the morphological evolution of a pair of cracks oriented parallel to the basal plane, originally 200 μm by 100 μm , and with a depth of 180 nm. The directions e and g are $[11\bar{2}0]$ and $[1\bar{1}00]$, respectively. Figure 4a illustrates the as-prepared crack geometry. The same two cracks are shown in Figure 4b after 60 min at 1800°C. The annular ring, evident in the cross section in Figure 3, is unstable to growth of sufficiently long wavelength perturbations. These perturbations cause a variation in the local crack opening displacement. A decrease in crack opening displacement increases the crack regression rate, and thus, points at which the annulus is thinner will recede more rapidly. This, coupled with longitudinal mass redistribution, causes what appears as local pinch-off of the annular ring. Once closure occurs, healing via intrusion is rapid; Figure 4c shows the interface after 90 min at 1800°C, and a considerable fraction of the original crack has been healed. Figure 4d shows the crack after 135 min total annealing time. At this stage, the crack contains several cylindrical segments. These are subject to Rayleigh instabilities. This later stage of healing has also been investigated using lithographically introduced features like those shown in Figure 2c [5]. The sequence presented is qualitatively consistent with the general observations summarized by Gupta [8].

A comparison between the healing behavior of the cracks in Figure 4, and those of other basal plane cracks of identical geometry and crystallography shows that the differences in healing behavior are related to the relative extents of crack regression before perturbation initiates. The results presented in Figure 4 illustrate a case in which there was a relatively greater propensity for pinch-off along the crack perimeter. Rim perturbation was dominant, resulting in more rapid healing. The relatively finer scale of pinch-off results in a finer-scale healed structure, and accelerates break-up into spherical pores. In other cases, crack regression was dominant. This leads to a reduced healing rate, and a much coarser healed structure.

Dramatic differences in morphological evolution can occur when the crystallography of the crack edges and faces is altered. One type of healing behavior for cracks oriented parallel to the prismatic plane is illustrated in Figure 5.¹ Figures 5a and 5b show two initially 120-nm deep cracks oriented parallel to the $(11\bar{2}0)$, after 5 and 210 minutes at 1800°C. Crack healing occurs via the growth of "pillars" or columns that ultimately connect the crack faces. During continued annealing at elevated temperature, the columns expand, and thereby heal considerable portions of the crack. This mode of healing has the potential to produce a significant increase in the healing rate relative to that due to crack regression alone, and provides a mechanism for healing cracks at positions away from the crack perimeter. Hickman and Evans have observed similar behavior in calcite [10].

The potential effects of grain face microstructure on crack healing became evident when the stability of flaws at single crystal-polycrystal interfaces was studied. If the flaw intersects multiple grains, grain boundary grooves will form. When the dominant transport mechanism for grooving is either surface diffusion [18,19] or volume diffusion [20], a shoulder or hillock develops adjacent to the groove, that extends above the original surface. These hillocks, if they become sufficiently large, can bridge the crack. This hillock-induced healing was observed, and bridging appeared imminent at other sites. The degree to which this mechanism can contribute to healing will be sensitive to the crack opening displacement variation with distance from the crack perimeter. The grain size will influence the spacing of the bridging sites, thus introducing a potential microstructural sensitivity to healing behavior.

All processing steps except for hot pressing are conducted in Class 100 clean rooms. Nonetheless, even with these precautions, occasional isolated contamination of the interface by airborne contaminants occurs. Figure 6 illustrates behavioral differences that we believe reflect the effects of inadvertent contamination of one crack (out of a total of 1080). The cracks shown are oriented parallel to the basal plane, and have an original depth of 180 nm. Figure 6a shows the as-prepared structure. After only 5 min at 1800°C, one of the cracks is essentially completely

¹ Not all prismatic plane flaws healed in this manner. Alternatively, pore channels formed parallel to the original crack front. The healing mechanism is distinct from that for basal plane cracks, and does not involve instabilities along the crack perimeter, but instead closure parallel to and away from the crack perimeter.

healed. After a total anneal time of 200 min, Figure 6c, the channels have broken up into isolated pores. The other crack has barely begun to heal. The healing rates of these two cracks are estimated to differ by two to three orders of magnitude.

Systematic studies utilizing doped or ion-implanted crystals may provide information on effects of crack face contaminants on transport during healing. Such studies could also provide useful insights on the role that these impurities play in affecting microstructural development during sintering [21]. It is conceivable that measurements of this type may also show the effects of anisotropic segregation [22] on processes whose kinetics are surface diffusion controlled.

Related Research Opportunities:

Our initial research objective has been to study several aspects of high temperature crack healing. However, the basic sample preparation procedures that were used lend themselves to several other applications.

Since it is possible to produce cracklike defects of controlled size and location, and to transfer them to internal interfaces, the technique appears to be an ideal means of fabricating standards for nondestructive testing methods, and should provide an interesting alternative to methods relying on the burn-out of latex spheres. Since lithography can be used to produce a sharp precrack at the interface as well as a controlled geometry pore array, it should be possible to study fracture in microdesigned porous materials. There are also a growing number of cases where simple components are bonded or joined to make a complex structure. The elimination of controlled-geometry flaws at bonded interfaces as a function of time, temperature, geometry, and applied pressure is amenable to investigation. A simple extension of the procedures described should allow the study of flaw elimination at ceramic-metal interfaces.

APPLICATION TO PORE-BOUNDARY INTERACTIONS

The following section summarizes our work on the application of photolithography to studies of pore-boundary interactions and pore-boundary separation in MgO-doped and undoped high purity alumina. A more detailed account of this work is available in references 1 and 3.

Background:

Significant advances in our understanding of sintering have resulted from modelling sintering as a competition between densification and coarsening processes [23,24], and investigating the competitive processes individually. Hot pressing provides a means of studying "pure" densification processes, and has proven helpful in identifying densification mechanisms [25].

An improved understanding of "pure" coarsening processes is also needed. Investigations of (grain) coarsening in a pore-free material are the most straightforward [26,27]. The characteristics of grain growth in porous compacts, specifically the nature of pore-boundary interactions during sintering, also have an important effect on microstructural evolution [28-33]; premature pore-boundary separation precludes complete densification during sintering.

Experimental analogues to hot pressing, capable of critically testing models of pore-boundary interactions, were lacking. As a result, experimental studies of pore drag have been limited to one of two approaches. The first is indirect, and relies on measurements of transport coefficients [5,34]. The second approach to studying pore drag yields only qualitative information [35].

It is desirable to have an experimental procedure amenable to controlled study of pore drag, which would allow determination of the critical condition for pore-grain boundary separation [36]. Ideally, this experiment would circumvent the problems associated with sintering studies, that is, it would provide a well-defined geometry, and it would eliminate competitive densification processes. The application of lithography to pore-boundary separation has led to the development of an experimental procedure that satisfies these restrictions, and provides a new tool for studying sintering, "controlled pore drag" experiments. Such controlled pore drag experiments, together with hot pressing experiments, allow study of the two idealized or limiting paths of microstructural evolution during sintering.

Experimental Considerations:

Controlled pore drag experiments rely on the production of controlled-geometry pore structures [2] at the interface between a large grain (single crystal sapphire) and a dense polycrystalline matrix. During subsequent heat treatments, the large "abnormal" grain consumes the adjoining polycrystalline material at a rate determined by the drag force exerted by the interfacial pore arrays. Pore-boundary separation occurs when the grain velocity exceeds the peak pore velocity. The important experimental variables are the polycrystal grain size and chemistry, and the pore size and spacing. Since the microstructure and chemistry of the polycrystal can be controlled, and pore size and spacing can be controlled precisely and varied easily via lithography, a wide range of conditions can be examined.

Controlled surface structures like those illustrated in Figure 2a, were generated on the basal plane of sapphire. The pore width was held constant at 3 μm , center-to-center pore spacings were varied (4, 6, 8, and 10 μm), and the pore depth was 0.24 μm . Pore arrays 20 pores wide and 1800 to 4000 pores long with interarray spacings of 200 μm were produced. Etched sapphire wafers were subsequently hot pressed against highly polished dense MgO-doped or undoped polycrystals which were fabricated using a two-stage hot pressing and hot isostatic pressing procedure, thus transferring the controlled pore structures from external to internal interfaces, as illustrated in Figure 7. Wide pore-free ligaments surrounding the pore arrays were used to almost completely suppress densification during hot pressing and annealing.

The microstructures of both undoped and 250 atomic ppm MgO-doped materials were uniform. Changes in grain size during annealing were determined using a linear intercept method. The migration rates of the sapphire-alumina interfaces were obtained by measuring boundary positions after various tempering times relative to the initial interface position as marked by lithographically introduced reference markers.

Results and Discussion:

The essential parameters in models of pore-boundary interactions are the grain boundary mobility, M_b , the pore mobility, M_p , and the areal density of pores. The mobilities can be assessed by measuring grain boundary and pore velocities under known driving forces; the areal density of pores is controlled using the procedures described previously.

The grain boundary velocity, V_b , was determined by monitoring the growth of unetched (pore-free) basal plane sapphire wafers into both doped and undoped dense alumina polycrystals. Results indicate a mobility of $1.8 \times 10^{10} \mu\text{m/Ns}$ for growth into undoped alumina at 1600°C, and a mobility of $2.5 \times 10^{10} \mu\text{m/Ns}$ for the doped material.

The critical condition for pore-boundary separation is determined by monitoring the growth of etched sapphire wafers with controlled geometry pore arrays into the same doped and undoped dense alumina polycrystals. Following an initial period during which the pore shape changes from that of a cracklike flaw to that of a more equiaxed intergranular pore, migration of the pore-laden interface commences. During migration, the pores either remain attached to the migrating interface, or under some conditions, the pores separate, and thereby become isolated in the growing single crystal sapphire wafer. Examples of pore-boundary separation in MgO-doped alumina at two different pore spacings are provided in Figure 8. The sample was annealed for a total of 15 h at 1600°C; separation occurred after ≈ 10 h of annealing.

Measurements of the migration kinetics include measurements of the pore velocity just prior to pore-boundary separation, and therefore velocities approaching the so-called peak pore velocity. When measurement errors are taken into account, the peak pore velocity, V_p , for undoped alumina is in the range of 0.8 to 2.2 $\mu\text{m/h}$, whereas the range for MgO-doped alumina is between 4.5 to 7.0 $\mu\text{m/h}$. Using a relationship derived by Hsueh et al. [28], the peak pore velocity can be related to the surface diffusivity. Our measured pore velocities yield estimates for the surface diffusivity of $0.62\text{--}1.70 \times 10^{-7} \text{ cm}^2/\text{s}$ for the undoped alumina, and $3.47\text{--}5.40 \times 10^{-7} \text{ cm}^2/\text{s}$ for the MgO-doped alumina, suggesting that the addition of 250 ppm MgO increases the surface diffusivity by a factor of 2 to 8 at 1600°C.

Substitution of the peak pore velocities into an expression describing pore-boundary separation in the case of constant center-to-center pore spacing [33], yields critical grain size-pore spacing coordinates at which separation initiates, and which thus divide a map of grain size and pore spacing into regions of pore separation and pore attachment. In the absence of any measurement error, a single line would divide the plot into attachment and separation regions. Figure 9 shows the correspondence between experimental results obtained for undoped (9a) and doped alumina (9b). The additional region, labelled B, reflects the uncertainty due to measurement error. For undoped alumina the correlation is particularly good. Further experimental work is necessary to better define the transition condition for MgO-doped alumina. However, given the uncertainty in values of parameters such as the interfacial energies and dihedral angles, which enter into the analysis, the results are encouraging.

Related Research Opportunities:

The results indicate that quantitative measurements of pore-boundary separation conditions are now possible. Since experiments can be conducted using both doped and undoped polycrystals, a direct comparison between theory and experiment is possible, and the effects of dopant additions can be assessed. It should be possible to clarify and quantify the effects of other dopants in alumina on pore-boundary separation conditions. More generally, since the technique can be applied to other systems, it may provide a general method for screening and assessing the effects of sintering additives on pore-boundary separation. When used in conjunction with other model experiments that provide information on impurity effects on lattice and grain boundary transport, a better understanding of impurity additions on microstructure evolution may evolve.

APPLICATION TO PORE COARSENING AND PORE ELIMINATION

During the final stage of sintering, vacancies are redistributed from pores to either larger pores (coarsening) or to the grain boundary (densification). In both cases, diffusion occurs through the lattice and along the grain boundary. When diffusion is the rate controlling process, a quantitative description of coarsening and densification requires that the lattice and grain boundary diffusivities be known. Alternatively, interfacial reactions may be rate limiting. Lithography provides a means of studying pore coarsening and pore elimination under highly controlled conditions [4].

Research Opportunities:

Figures 2a and 2b establish the ability to produce monodispersed and bimodal surface features that, after bonding, yield monodispersed and bimodal pore arrays. As has been shown for other cases, these features can be transferred to an internal interface. The surface crystallography and the grain boundary misorientation can be controlled. Moreover, the impurity content of the crystals can also be varied. This combination of features provides numerous research opportunities [4].

For the sake of brevity, only diffusion controlled processes will be considered. First, the ability to vary the "volume" fraction, size, size distribution, and spatial distribution of interfacial pores, and to systematically vary each of these parameters allows unprecedented control over the topological characteristics that influence the coarsening and densification rates. Second, through a systematic increase in boundary misorientation, from zero degree misorientation upward, it should be possible to separate the contributions of lattice and grain boundary transport to coarsening and densification. When the design of the interfacial structure permits pore elimination, effects of an applied pressure on pore elimination rates can be assessed. Finally, since the experimental technique can be applied equally well to doped and undoped material, comparisons between the behavior of doped and undoped materials under otherwise identical experimental conditions offer the possibility of clearly identifying the effects of dopants on grain boundary and lattice transport coefficients.

EXTENSIONS AND REFINEMENTS

There appear to be two logical avenues for extending the range of processes that can be investigated. The first entails increasing pattern complexity. The surface patterns generated to date have consisted of discrete entities, isolated pores, channels, cracks, etc.. The generation of controlled geometry two-dimensional continuous patterns affords new experimental opportunities. Pore networks in unfired or partially densified ceramics can be mimicked. Removal of a phase such as a liquid or a polymer can be examined; Cima and co-workers have applied the method to studies of binder removal. Alternatively, the infiltration characteristics of porous networks by a vapor or a liquid metal can be investigated. Finally, the coarsening or densification behavior of such pore networks can be studied.

The second avenue for extension and refinement entails a decrease in the scale of the structures that are studied. There is a continuous effort to reduce the scale of features in the microelectronics industry, which drives the development of submicron lithographies. State of the art microfabrication laboratories include facilities for lithographies that are capable of producing 0.4 μm width features. When even finer features are of interest, electron beam lithography can be applied. The National Nanofabrication Facility at Cornell University has facilities that provide 25 nm resolution [38]. Consequently, simulation and study of nanoscale microdesigned interfacial structures should be possible.

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FIGURES

- Figure 1 Schematic illustration of sample preparation procedure: a) coated substrate, b) selective exposure through a mask, c) ion beam etching of substrate and patterned photoresist, d) etched photoresist and substrate, and e) bonding.
- Figure 2 SEM micrographs of accessible surface structures: a) monomodal arrays used for pore-boundary separation studies, b) bimodal arrays for pore coarsening studies, c) channel-like features for studying late stage crack healing and Rayleigh instabilities, and d) nonequilibrium shapes for studying facet development and surface energy anisotropy.
- Figure 3 SEM micrograph of a cracklike flaw lying parallel to the basal plane, viewed in a cross section taken perpendicular to the crack plane after 10 min at 1800°C. $e = [11\bar{2}0]$ and $f = [0001]$
- Figure 4 Optical micrographs of a pair of 100 μm by 200 μm by 180 nm deep flaws at 1800°C after a) 0 min, b) 60 min, c) 90 min, and d) 135 min. The directions e and g are $[11\bar{2}0]$ and $[1\bar{1}00]$, respectively. Edge perturbation dominates and leads to a finer scale healed structure.
- Figure 5 Two cracks, initially 120 nm deep, oriented parallel to the $(11\bar{2}0)$, after a) 5 and b) 210 minutes at 1800°C.
- Figure 6 Effect of inadvertent contamination on healing rates of two otherwise identical cracks etched into the basal plane of sapphire. Crack evolution after a) 0 min, b) 5 min, and c) 200 min at 1800°C.
- Figure 7 Tailored interface with varying pore density. Pore spacing: a) 4 μm , b) 6 μm , c) 8 μm , and d) 10 μm .
- Figure 8 Illustration of pore-boundary separation in MgO-doped alumina in samples with two different pore spacings.
- Figure 9 Calculated and observed separation conditions for a) undoped and b) 250 atomic ppm MgO-doped alumina at 1600°C.

Sample Preparation Procedure

XBL 894-1445

Figure 1

XBB 894-2696

Figure 2

XBB 883-2636

Figure 3

XBB 885-4842

Figure 4

XBB 885-4843

Figure 5

XBB 883-2635

Figure 6

Tailored interface with varying pore density. Pore spacing: a) $4\mu m$, b) $6\mu m$, c) $8\mu m$, d) $10\mu m$.

XBB 882-742

Figure 7

XBB 882-746

Figure 8

XBL 8810-3548 A

Figure 9

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