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PLASTIC DEFORMATION OF CERAMIC MATERIALS

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ABSTRACT

Complexity of the characters of ceramic materials enhances difficulties of evaluating effect of various parameters on the plastic behavior of the materials. Grain and grain boundary processes contribute to plastic strain. Factors of greatest uncertainty are the nature and effect of the grain boundaries and adjoining regions, and of the pores. Equally important is the need of a science of ceramic processing so that specimens with designed characters (microstructures) could be fabricated for controlled mechanical behavior studies.

INTRODUCTION

Ceramic materials consist of nonmetallic inorganic compositions ranging from completely crystalline to completely glassy structures. Polycrystalline ceramics essentially free of a secondary glass phase are of greatest interest for specialized industrial applications. Their mechanical behavior is dependent upon their character. The presence of any impurities, second phase or disordered structure along grain boundaries is particularly significant. Most of these materials also contain a pore phase which further affects their behavior.

In contrast to metals most polycrystalline ceramics can be deformed plastically only at elevated temperatures when diffusion-controlled deformation mechanisms usually become active in addition to any slip. Because of the complexity of the characters, many material parameters can affect deformation behavior either singly or cooperatively. These are often difficult to identify, characterize and control. In many cases experimental observations are too limited to provide complete understanding of the deformation mechanism and the effect of material character. Some of the questions and uncertainties with respect to these aspects based on our current understanding are presented in the following paragraphs using data for several oxides as illustrative examples.

DEFORMATION MECHANISMS

Plastic strains exhibited by a material are produced by grain processes and grain boundary processes, either independently or cooperatively. Grain processes include dislocation glide, dislocation climb, dislocation glide and climb, kinking and twinning. Grain boundary processes include diffusional and viscous mechanisms, grain boundary sliding and intergranular separation. Grain boundary migration may affect deformation but will not produce any strain by itself.

1. Grain Processes

In order to realize ideal polycrystalline ductility, as shown by von Mises,¹ slip must take place on a large scale and homogeneously on 5 independent slip systems in every grain with retainment of grain boundary integrity. In cubic ceramics of NaCl (e.g. MgO) or CaF₂ (e.g. UO₂) structure, 5 independent slip systems are provided by two families of slip planes (i.e. {110} and {100}). These planes become active at different critical resolved shear stresses, the difference increasing with decrease in temperature.^{2,3} Unlike cubic metals, therefore, plastic anisotropy exists. In addition, intersection difficulties exist both at 90° and 120°, the latter being more difficult.⁴ Thus, ductility may still be limited because of these factors even when all 5 independent systems are provided. Also, the fact that certain slip planes (e.g. {100} in the case of MgO) are also cleavage planes is significant. All of these points warrant further investigation as to their effect on deformation.

Although it is acknowledged that cross-slip can in effect reduce the required number of slip systems,⁵ it is not known to what extent.

Cross-slip is likely to involve a change of slip planes between easy and difficult ones ($\{110\}$ and $\{100\}$ planes in the case of NaCl structure). The effectiveness of this process may still be dependent on the slip anisotropy. The degree of correlation is unknown.

It has been shown recently by Nabarro⁶ and subsequently by Groves and Kelly⁷ that climb by itself or in conjunction with glide, can produce a strain component. With suitable combinations of primary slip and climb systems, some ceramics apparently can satisfy the von Mises requirement without activating secondary slip. While Nabarro's dislocation climb mechanism may be operative in certain materials,⁷ Groves and Kelly's mechanism has not been identified, probably because the latter mechanism has not been formulated quantitatively. Only the glide contribution has been emphasized in a recent study on MgO with the climb contribution limited to an indirect effect on dislocation density.⁸

The attractive feature of such mechanisms as cross-slip and climb is the resulting relaxation of the von Mises requirement. There is an indication, however, that this does not happen during creep of a dense Al_2O_3 polycrystal. As shown in Fig. 1, creep occurs with a stress exponent of 1 or 2 and at stresses exceeding steady flow stresses for basal slip.¹⁰ In this case, the combination of basal and prismatic slip does not provide 5 independent systems; pyramidal slip alone can provide them¹¹ but they would become activated at considerably higher stresses.^{12,13} Questions as to whether the potential relaxing mechanisms are ineffective, whether the amount of basal slip is insufficient to activate pyramidal slip, or whether even the basal slip system is inactivated because of impurity locking or accommodation of stress concentration by a

diffusional process remain to be answered.

Limited dislocation motion in grains will cause pile-ups at grain boundaries accompanied by stress concentrations which can be relieved by either microcracking or secondary slip depending on the strength of the grain boundaries. The occurrence of microcracking at low temperatures usually leads to fracture before any macroscopic yielding is observed.

If microcracking does not occur, then stress concentrations due to primary slip can reach a level sufficiently high to locally activate the secondary slip system. Hence, the von Mises requirement would be fulfilled, at least near grain boundaries. As grain size decreases, a greater fraction of the grain volume would fulfill the requirement. On this basis, a decrease in grain size would be accompanied by increasing ductility and decreasing yield stress. However, Stokes¹⁴ found an increased ductility for a larger grain size in polycrystalline NaCl tested at an elevated temperature. It was rationalized that as the grain size increased the compatibility requirements in the vicinity of the grain boundary had relatively less influence over the remaining volume of material, permitting a greater amount of plastic strain. Significance of the extent of localized multiple slip, therefore, deserves further attention.

Twinning has been observed to occur on various crystallographic planes in non-cubic crystals such as Al_2O_3 . It has been invoked to explain slightly nonviscous creep behavior of polycrystalline Al_2O_3 .¹⁵ Some authors¹⁶ believe that twinning can produce strain and contribute to ductility in hexagonal metals. Since twinning occurs only in one direction, however, it seems that its effectiveness in reducing the

required number of slip systems is likely to be limited. Furthermore, twinning can cause premature fracture because of the associated stress concentrations unless they are accommodated in some way by surrounding material. Hence, the combined effect of twinning presently seems unpredictable.

Kinking has been observed in cubic single crystals such as MgO ¹⁷ and CsBr .¹⁸ Cooperative kinking could be a means of producing strains as pointed out by Washburn and Parker.¹⁹ Its significance in polycrystalline deformation, however, has not been established experimentally.

2. Grain Boundary Processes

It appears that the nature of the grain boundary is extremely important in all deformation processes of ceramic materials. In the discussion on grain processes it was assumed that dislocations had no difficulty in nucleating and propagating which involves displacements on the surfaces or interfaces of the grains. Actually, a fundamental question concerns itself with whether dislocations have difficulties of being nucleated at grain boundaries and, if they are nucleated at an internal site such as a pore, whether they can penetrate grain boundaries. There are indications that under certain conditions grain boundaries are inert and also sufficiently strong to resist any microcracking and thus allow the specimen to realize high strength (specimen G in Fig. 2(a) may represent this condition).

Under certain conditions the grain boundary may provide a diffusion path resulting in a Coble type of creep²⁰ wherein mass transport occurs from grain boundary regions of compression to regions of tension. The same type of deformation can also be realized by the Nabarro-Herring

mechanism^{21,22} which represents mass transport through the grains with the grain boundaries acting as a source and sink for vacancies. Which of these mechanisms, both with a stress exponent of 1 in creep, actually occurs under given conditions is not clear. On the other hand, there have been reports that grain boundaries in UO_2 ²³ and Al_2O_3 ²⁴ may not be a perfect source and sink for vacancies when the applied stress is very low. Certain parameters such as test temperature,²³ stoichiometry^{25a} and grain size^{25b} have been found to affect the threshold stress below which creep is controlled by the effectiveness of grain boundaries as a source and sink for vacancies. However, the specific nature of grain boundaries susceptible to such behavior is unknown. In addition, grain size dependence of activation energy for diffusional creep of MgO as reported by Passmore et al.²⁶ remains unexplained.

Grain boundary sliding (GBS) which represents relative movement of two adjoining grains along a grain boundary is a common occurrence during high-temperature deformation. As a result of this phenomenon, stress concentrations develop at triple points and at irregularities along the boundary. These stress concentrations must be dissipated by accommodation processes, or else a boundary will separate. Therefore, the extent of GBS contribution to total strain is likely to depend on the ease of accommodation. Kocks²⁷ has pointed out that GBS cannot provide an independent component of the microscopic strain unless whole sheets of grains in the entire cross section slide over each other. Nevertheless, Langdon²⁸ has proposed that GBS might be an independent deformation mechanism capable of accounting for the total strain if complete coherency of grain boundaries is not a rigid requirement.

Consequently, a question remains to be resolved as to whether GBS contributions should increase, and under what conditions, as other deformation mechanisms become less effective.

GBS involving neither grain deformation nor triple-point accommodation has been visualized by Cannon and Nix²⁹ as sliding of aligned grains in groups. Although such configurations might lead to a large ductility, how such an alignment could be attained is not at all clear.

Effects of GBS and GBS-associated processes also require better understanding. For instance, GBS might change the structure of a grain boundary so that the boundary can be penetrated by dislocations from the grains. GBS may cause rotation of grains such that the grains unfavorably oriented for slip will be brought into a more favorable orientation although occurrence of the opposite effect is also conceivable.

As frequently observed in metals, GBS and grain boundary migration can occur simultaneously.³⁰ Grain boundary migration could relieve stress concentrations due to GBS, and hence inhibit intergranular cracking as suggested for hyperstoichiometric UO_2 .³¹ A complicating factor is that other investigators have observed that nonstoichiometric UO_2 was more susceptible to GBS and to creep damage.³² The apparent contradiction here illustrates the lack of understanding of the complex relationships between composition and sliding with migration of grain boundaries. Another complicating factor is that rumpled grain boundaries caused by localized migration can provide a barrier for GBS³³ rather than a means of accommodation. Grain boundary migration can also

facilitate the transfer of slip across the boundary.³⁴ Significance of such effects need further detailed evaluation.

Depending on test and perhaps material conditions, intergranular separation (IGS) occurs in the form of cavities at triple points and grain boundaries, and of intergranular cracks whose maximum length is the grain size, as a mechanism to relieve stress concentrations associated with dislocation pileup or grain boundary sliding. This process can give rise to an additional strain accompanied by increased volume, while it is not immediately clear how grain deformation would be affected by IGS. In creep of stabilized ZrO_2 , Evans³⁵ suggested that strains and a high stress exponent (~ 6) for steady creep rates could be attributed to IGS. Similarly, Morrell and Ashbee³⁶ explained increasing stress exponents (up to 6) in glass-ceramics in terms of formation of voids within the glassy phase between the crystals. On the other hand, it has been also shown that high values of stress- and temperature-dependency can be reduced if internal stress is taken into account.³⁷ Uncertainties exist between these possibilities.

At least in tension, intergranular cavities may increase the rate of dislocation creep by serving as vacancy sinks and hence increase the rate of dislocation climb.³⁸ This possibility may add to the uncertainties of understanding deformation of porous materials. Apparently, under certain conditions, intergranular separations are stable and do not cause immediate fracture nor tertiary creep at high temperatures.^{35,36,39,40} It is thus of interest to determine why IGS can remain stable and how steady flow can be maintained with continuing IGS.

MATERIAL CHARACTER EFFECTS

Material characteristics play a role in dictating what deformation mechanism is active under imposed testing conditions. It thus becomes significant to explore the effect of several recognized parameters of character on the plastic deformation of ceramic materials.

1. Composition of Grains

Attempts have been made, notably by Cannon and Sherby,⁴¹ to correlate composition in terms of ionic size ratios with dislocation creep mechanisms. The concept is that if the anion/cation ratio is large, the charge on the dislocation is large, limiting the ease of dislocation glide. Thus, it was predicted that creep of MgO would exhibit a stress exponent of 3. However, a range of exponents has actually been reported, as seen in Fig. 2(b), suggesting that in a polycrystalline material other factors are important as well. In the case of dislocation glide it is suggested that the nature of chemical bonding may be a critical material parameter. It is significant that polycrystalline AgCl is very ductile and exhibits wavy slip, even at room temperature.⁴² Academic studies of this nature are extremely worthwhile since they contribute to an understanding of the deformation processes on an atomistic level.

Of greater practical significance is the effect of impurities or additives on single crystal or grain behavior. Both hardening⁴³ and softening⁴⁴ have been observed in single crystals deformed at high temperatures, depending on the combination of cation impurity, solvent and testing environment. Under creep conditions, temperature-dependency⁴⁵ and/or stress-dependency⁴⁶ can be affected. Information on the effects

of anion impurities is extremely meager. In general, further systematic studies are in order with an objective of identifying additives that would reduce plastic anisotropy, increase ease of cross-slip and ease of slip interpenetration, and reduce cleavage cracking without loss of strength.

2. Geometric Character Parameters

Pores are a significant feature of practically all polycrystalline ceramic material characters. There are very few ceramic materials free of pores that are also free of a glassy phase which was present as a liquid during firing. Pores can reduce the load-carrying cross section area, cause local stress concentrations and lower elastic moduli. Thermodynamically stable cavities can absorb vacancies.³⁷ Dislocations may be not only nucleated from,⁴⁷ but also attracted to,⁴⁸ pores; they may be hindered by intragranular pores.^{49,50} While pores can nucleate cracks, it has been proposed that they can interfere with crack propagation^{51,52} and serve as accommodation sites.⁵³ Yet, another example of opposite effects is that intergranular pores might facilitate GBS but suppress grain boundary migration.⁵⁴ However, as mentioned previously, suppression of migration might be accompanied by inhibition of sliding as long as the former process is one of the stress-relieving mechanisms for the latter. Details of these effects can become even more complicated when the wide range of size, shape and distribution of pores is taken into account.

Grain size and uniformity are well recognized character parameters in determining mechanical behavior in ceramics as well as metals. Generally, the same relations are also observed. There are, however, some

apparently inconsistent relationships. Creep resistance of UO_2 has been reported by Burton et al.^{25b} to obey a Hall-Petch type of relation at high stresses with an unusually high stress exponent of ~11. Occurrence of dislocation creep should have exhibited no dependence on the grain size with a lower stress exponent. In creep tests of doped Al_2O_3 , grain size independence was observed with a stress exponent slightly greater than unity⁴⁰ although grain size dependence would have been expected with such an exponent. Intergranular separation might account for this behavior; the evidence, however, is not yet conclusive.

3. Grain Boundary Structures and Compositions

Grain boundaries are the most difficult to characterize both in terms of structure and composition. A considerable amount of effort, however, is being expended in this direction with the availability of new research tools for the investigation of surfaces. As yet, their effect on the plastic deformation of polycrystalline ceramics is most difficult to analyze because of the complexity of the distribution of impurities and additives and because of the multiplicity of the operative deformation processes.

Generally, relatively minor amounts of additives (as impurities or intentional) tend to concentrate in the grain boundaries and/or in the portion of the grain adjacent to the grain boundaries. If the additions exceed the solubility limits, they can form films or definitely identifiable secondary phases (generally glass) along the grain boundaries.

Impurities of certain kinds could affect cohesive strength, diffusivity along the grain boundaries, and the migration ability of the

grain boundary during mechanical testing. If the impurities also segregate in the zone adjacent to grain boundaries, the hardness of the specimen could change because of changing resistance to dislocation motion. On the other hand, impurities might also affect GBS. There are many uncertainties concerning the net effect because of expected complex interactions between multiple processes. Thus, there may be synergistic effects, but also some events may tend to counteract each other.

Most ceramic materials contain some glassy phase. Its effect on mechanical behavior is not always predictable, probably because of inadequate characterization of the glassy phase and incomplete understanding of its behavior. Below the glass transformation temperature glass behaves as a brittle solid and above, as a liquid whose viscosity is dependent on composition and temperature. There is evidence, however, that the glass does not always deform by viscous shear flow above the glass transformation temperature.⁵⁷ Furthermore, the degree of connectivity of both phases should be critical in determining the behavior of the material, but this understanding is just developing.

STRESS-STRAIN AND CREEP BEHAVIOR

Diversity of compressive stress-strain and creep behavior at 1200°C of polycrystalline MgO specimens with comparable densities and grain sizes but fabricated in different ways (Table I) is shown in Figs. 2(a) and 2(b). They are presented as examples to illustrate the above-mentioned subtleties and uncertainties which make generalizations difficult and emphasize the importance of character.

Figure 2(a) shows that strength and ductility are very sensitive to processing variables. Comparison between pairs of specimens prepared under comparable processing conditions except for annealing procedure (A vs. B and III vs. II) indicates that annealing in air (specimens A and III) following hot-pressing is essential for improved ductility. It is reasonable to attribute this difference to "clean" grain boundaries and hence strong intergranular cohesion which permitted accommodation mechanisms to take place. The limited ductility and possibly yield drop of specimen I in comparison with Specimen III is in accord with this suggestion, since grain boundaries in I are presumably contaminated by residual Li and/or F ions. The yield drop, however, did not occur in a similar specimen F when the test was run at a constant loading rate. The ductile specimens A and E are also characterized by a distribution of fine pores inside the grains. Even though the strong grain boundaries appear to be necessary, the presence of the fine pores may play a critical role in the realization of homogeneous deformation in view of the possibilities discussed earlier. Consequently, the origin of the observed ductility is not completely clear.

Specimens A, III and G form an interesting sequence. The comparatively low flow stresses for specimen A can be associated with its clean grain boundaries which could allow the development of sufficiently high stress concentrations for local activation of secondary slip systems. The higher flow stress and fracture strain for III is not understood; it could be due either to differences in processing or method of loading (constant strain rate vs. constant stress rate). Specimen G exhibited the highest strength with some ductility; it also showed high strength at 1000°C but complete brittleness. Presence of strong grain boundaries and immobilization of dislocations at grain boundaries are thus indicated. This specimen also had relatively large pores at triple points which could have participated in some accommodation process at 1200°C. A general understanding of the critical parameters can thus be developed, but a better and more quantitative understanding is not possible until better characterization techniques for the grain boundaries become available.

Curves C and D in Fig. 2(a) are for specimens with 5% glass but with a ratio of grain-grain to total contact areas of 0.5 and 0.3, respectively. Under the indicated conditions the glass apparently does not flow by shear and the specimen behaves in a brittle manner. At higher temperatures it is expected that the glass would deform in a viscous manner and the strength of specimen C would become greater than that of D with an increase in ductility. Carefully controlled studies are needed for a more specific analysis.

Figure 2(b) shows that creep rates at a given stress and the stress exponent (indicative of the operative mechanism) also exhibit a

dependence on the character of the material as affected by the heat treatment. The presence of high stress exponents in comparison with those for Al_2O_3 in Fig. 1 suggests that slip anisotropy for MgO is less than that for Al_2O_3 . Although specimen A is weaker than B in the stress-strain test, its creep resistance is greater. Its high creep resistance together with high stress exponent indicates a dislocation mechanism which is consistent with its plastic behavior in the stress-strain test. Extending Ashby's proposal that there should be a critical stress for creep of porous material,⁶² it may be argued that distribution of pore sizes gives rise to a range of critical stresses, or equivalently some high stress exponent. On the other hand, the stress exponent of ~ 2 for specimen B agrees with Langdon's GBS model; it points to the fact that possible grain boundary modifications by (carbonaceous) impurities introduced in processing now play a dominant role. This behavior is also consistent with the brittle nature of its stress-strain curve (Fig. 2a) in comparison with specimen A.

The creep behavior of specimen D is similar to that of a specimen with no glass deforming by some dislocation mechanism. In this case the glassy phase appears to support the stress and still conform with the deformation of the MgO framework. Further experimental and theoretical work is needed since detailed interpretations are still open to question.

While specimens F' and K' behaved similarly when crept at lower stresses, the latter specimen exhibited a somewhat higher stress exponent (4.4 vs. 3.3) at high stresses. Since both specimens are normally comparable in nature, the observed disparity remains to be explained. It is also of interest why the specimen K' shows a creep resistance

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similar to that of specimen A at equivalent high stresses despite the presumed difference in grain boundary nature.

CONCLUSIONS

A review of reported studies indicates that an understanding of the plastic deformation of polycrystalline ceramics is developing. Because of the complexity of ceramic materials extensive specific studies are still needed to develop more quantitative relationships. The feature of greatest uncertainty is the nature of the grain boundary itself and of the regions adjacent to the grain boundaries, and their response to various stress conditions over a range of temperatures. Another area of uncertainty is the role that pores play in plastic deformation processes. Detailed studies are hampered because of difficulties in isolating a given parameter for determining its role in plastic deformation. A better understanding requires further development of a science of ceramic processing so that specimens with designed characters could be fabricated, and of techniques for fully characterizing the specimens.

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Table I. Character of Polycrystalline MgO Specimens

(Legend for Figs. 2(a) and (b))

Spec. No.	Fabrication	Density (% theoretical)	Grain Size (μm)	Reference
A	hot-pressed and annealed in air	99.2	17	55
B	hot-pressed	99.2	13	55
C*	hot-pressed and annealed in air	98.5	30	56
D*	hot-pressed with extrusion	98.4	31	56,57
E	sintered	>98.9	25	58
F	hot-pressed with LiF additive and annealed in air	~100	12	53
F'	hot-pressed with LiF additive and annealed in air	~100	12-52	59
G	sintered	>98.4	20	53
K'	hot-pressed with LiF additive and annealed in air	~100	13.60	60
I	hot-pressed with LiF additive and annealed in air	~100	26	60

Table I. (cont.)

Spec. No.	Fabrication	Density (% theoretical)	Grain Size (μm)	Reference
II	ball-milled, hot-pressed, and annealed in vacuum within a graphite die	98.3	30	61
III	ball-milled, hot-pressed, and annealed in air	98.3	28	61

Note. * = 95% MgO + 5% CaMgSiO₄

FIGURE CAPTIONS

Fig. 1. Steady-state creep rate vs. tensile stress for a polycrystalline alumina (Ref. 9) in comparison with sapphire single crystals oriented for basal slip (Ref. 10) and pyramidal slip on $\{1\bar{1}02\}$ planes (stress exponent of ~ 3 : Ref. 12) or on $\{20\bar{2}1\}$ planes (stress exponent of 6.9 : Ref. 13).

Fig. 2. (a) Stress-strain curves for various polycrystalline MgO specimens (see Table 1) at constant loading rates (A through G) and at a constant strain rate (I through III); fracture occurred at the end of each curve except for specimens E, F., and G.

(b) Steady-state creep rate vs. compressive stress for various polycrystalline MgO specimens (see Table 1).

Figure 1

XBL764-5375

Figure 2a

XBL764-5373

Figure 2b

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