

Report for DE-FG02-87ER45302

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II. BACKGROUND-RESEARCH ACCOMPLISHMENT OF THE PRIOR DOE/BES GRANT

"Fundamental Alloy Design of Oxide Ceramics and Their Composites"

May 1, 1990-August 31, 1992

During the above grant period, Professor Chen has been supported by DOE at a budget of around \$90,000/yr. The main research has been on the microstructural development of oxide ceramics. In addition, several projects which were continued from the previous grant period of May 1, 1987-April 30, 1990, were also completed. The following are the titles of publications dated May 1, 1990-August 31, 1992, in reverse chronological order. Copies of these publications are attached as Appendices. Their abstracts are given below in the subsections 2.1-2.12.

1. P.L. Chen and I-W. Chen, "Reactive CeO₂ Powders by Homogeneous Precipitation Method," submitted for publication to *J. of American Ceramic Society* (1992).
2. L.A. Xue and I-W. Chen, "A New SiC Whisker Reinforced Lithium Aluminosilicate Composite," submitted for publication to *J. of American Ceramic Society* (1992).
3. I-W. Chen, "Solute Drag on Grain Boundary in Ionic Solids—The Space Charge Effect," to be published in Design and Control of Grain Boundary Properties in Ceramics, Eds., K. Ishizaki and K. Nihara, Elsevier Publications, New York (1992).
4. P.L. Chen and I-W. Chen, "In-situ Alumina/Aluminate Platelet Composites," accepted for publication, *J. of American Ceramic Society* (1992).
5. X. Wu and I-W. Chen, "Exaggerated Texture and Grain Growth of a Superplastic Silicon Nitride," accepted for publication, *J. of American Ceramic Society* (1991).

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6. X. Wu and I-W. Chen, "Hot Extrusion of Ceramics," accepted for publication, *J. of American Ceramic Society* (1992).
7. L.A. Xue, K. Meyer and I-W. Chen, "Control of Grain Boundary Pinning in $\text{Al}_2\text{O}_3/\text{ZrO}_2$ Composites with $\text{Ce}^{3+}/\text{Ce}^{4+}$ Doping," *J. of American Ceramic Society*, 75 [4] 822-29 (1992).
8. I-W. Chen, "Superplastic Forming of Ceramic Composites," in Advanced Composite Materials, Ed. M. Sacks, *Ceramic Transactions*, 19, 695-706 (1991).
9. G.N. Hassold, I-W. Chen and D.J. Srolovitz, "Computer Simulation of Final Stage Sintering I: Model, Kinetics and Microstructure," *J. of American Ceramic Society*, 73 [10] 2857-64 (1990).
10. I-W. Chen, G.N. Hassold and D.J. Srolovitz, "Computer Simulation of Final Stage Sintering II: Influence of Initial Pore Size," *J. of American Ceramic Society*, 73 [10] 2865-72 (1990).
11. I-W. Chen and L.A. Xue, "Development of Superplastic Structural Ceramics," *J. of American Ceramic Society*, 73 [9] 2585-2609 (1990).
12. C.K. Yoon and I-W. Chen, "Superplastic Flow of Two-Phase Ceramics Containing Rigid Inclusions—Zirconia/Mullite Composites," *J. of American Ceramic Society*, 73 [6] 1555-65 (1990).

2.1 Reactive CeO_2 Powders by Homogeneous Precipitation Method

CeO_2 powders have been prepared by aging a cerium(III) nitrate solution in the presence of hexamethylenetetramine. Oxidation of Ce^{3+} occurs in the precipitate and the wet precipitate is identified as crystallized CeO_2 before any heat treatment. The cold pressed powders can be sintered to full density at temperatures as low as 1250°C in just 6 min. Moreover, the sinterability of the powders is insensitive to the calcination temperatures, particle size, or green density. The powders calcined at 850°C with a crystallite size of 600\AA have a sinterability as good as the powders calcined at 450°C with a crystallite size of 145\AA . The mechanisms for direct CeO_2 precipitation and its relation to the excellent sinterability are discussed.

2.2 A New SiC Whisker Reinforced Lithium Aluminosilicate Composite

The glass ceramic matrix of the well-known lithium aluminosilicate (LAS)/ SiC composite is usually formulated near the spodumene composition. We report here a new composition which is rich in alumina (78wt%) and lean in silica (21wt%) and lithia (1 wt%). This formulation offers a

new option of converting the glass ceramic matrix to a mullite/alumina matrix upon annealing above 1400°C, and hence better creep resistance and other high temperature mechanical properties. Using a transient phase processing method we developed previously for the superplastic forming of mullite, we are able to hot-press a composite containing 30 vol% SiC whiskers at ~1350°C to achieve full density. Flexural strength measurements up to 1400°C have confirmed the improved high temperature strength and creep resistance over conventional LAS. The fracture toughness is also higher than that of LAS. The results suggest that the new composition may be chosen as a better candidate matrix for SiC fiber reinforced composites.

2.3 Solute Drag on Grain Boundary in Ionic Solids—The Space Charge Effect

Solute drag involving space charge interactions between aliovalent dopants and counterions near a grain boundary is evaluated. We use a lattice model to describe site competition between solutes and host ions, which naturally leads to saturated solute segregation at the grain boundary at higher concentrations. In such a limit, the screening distance is nearly concentration independent, and the grain boundary mobility goes through a minimum at an intermediate concentration. In the dilute limit, the mobility decreases monotonically with solute concentration, and a slower diffusing cation dopant of a larger effective charge provides a larger drag. Recent grain growth data of ZrO_2 and CeO_2 are in support of the above model.

2.4 *In-situ* Alumina/Aluminate Platelet Composites

Alumina composites containing *in-situ*-formed hexaluminate ($LaAl_{11}O_{18}$, $LaMgAl_{11}O_{19}$, $SrAl_{12}O_{19}$, and $Mg_2NaAl_{15}O_{25}$) platelets can be pressureless-sintered to high density. The grain morphology of the aluminates can be controlled by composition. A peak toughness 50% higher than that of Al_2O_3 is obtained at 30 v/o aluminates, with a modest reduction (10%) in hardness and Young's modulus. Although crack-bridging by aluminate platelets is apparently operating, the maximum toughness is intrinsically limited by the low cohesive strength of these layer compounds.

2.5 Exaggerated Texture and Grain Growth of a Superplastic Silicon Nitride

A fine-grained superplastic β' -silicon nitride solid solution (SiAlON) was found to develop elongated grains and a pronounced texture during tensile deformation at 1550°C. The texture development is well described by a geometrical model of grain rotation in accordance with the strain field. Once aligned, grains can then grow with little constraint due to impingement and often coalesce into each other. With the above microstructural development, the stress-strain curve displayed unusually strong strain hardening characteristics due to a fiber-reinforcement effect of the aligned silicon nitride grains on the glass-containing matrix. By extending the rheological model of Chen and Yoon and considering these microstructural evolutions, we are able to simulate the deformation behavior.

2.6 Hot Extrusion of Ceramics

A solid $\text{Bi}_2(\text{Sr,Ca})_3\text{Cu}_2\text{O}_{8+x}$ ceramic has been hot extruded through conical dies. The product wires have a highly textured crystallographic structure with the basal plane aligned along the wire axis. The mechanics of the extrusion process has been studied both theoretically and experimentally with the die semi-angle from 30° to 60°, the extrusion ratio from 4 to 9, the temperature range from 775°C to 825°C and the outlet extrusion speeds from 0.002 to 0.8 mm/s. The tool friction coefficient is also estimated. A simple analytical model has been developed using a combined slab method and upper bound method for a power-law creeping, strain rate sensitive material. The model predictions are in good agreement with the experimental results.

2.7 Control of Grain Boundary Pinning in $\text{Al}_2\text{O}_3/\text{ZrO}_2$ Composites with $\text{Ce}^{3+}/\text{Ce}^{4+}$ Doping

The control of the microstructure of Ce-doped $\text{Al}_2\text{O}_3/\text{ZrO}_2$ composites by the valence change of cerium ion has been demonstrated. Two distinctively different types of microstructure, large Al_2O_3 grains with intragranular ZrO_2 particles and small Al_2O_3 grains with intergranular ZrO_2 particles, can be obtained under identical pre-sintering processing conditions. At doping levels greater than ~3 mol% with respect to ZrO_2 , Ce^{3+} raises the alumina grain boundary to zirconia

particle mobility ratio. This causes the breakaway of grain boundary from particles and the first type of microstructure. On the other hand, Ce^{4+} causes no breakaway and produces a normal intergranular ZrO_2 distribution. The dramatic effect of Ce^{3+} on the relative mobility ratio is found to be associated with fluxing of the glassy boundary phase, and is likewise observed for other large trivalent cation dopants. The ZrO_2 second phase acts as a scavenger for these trivalent cations, provided their solubility limit in ZrO_2 is not exceeded.

2.8 Superplastic Forming of Ceramic Composites

Ceramic composites offer the special advantage of microstructural stability during large strain superplastic deformation. They tend to have both a higher flow stress and local stress around reinforcing particles, especially when the aspect ratio of the inclusions is high. Thus, only equiaxed ceramic composites are amenable to superplastic forming at the present time.

2.9 Computer Simulation of Final Stage Sintering I: Model, Kinetics and Microstructure

A Monte Carlo model for simulating final-stage sintering has been developed. This model incorporates realistic microstructural features (grains and pores), variable surface diffusivity, grain-boundary diffusivity, and grain-boundary mobility. A preliminary study of a periodic array of pores has shown that the simulation procedure accurately reproduces theoretically predicted sintering kinetics under the restricted set of assumptions. Studies on more realistic final-stage sintering microstructure show that the evolution observed in the simulation closely resembles microstructures of real sintered materials over a wide range of diffusivity, initial porosity, and initial pore sizes. Pore shrinkage, grain growth, pore breakaway, and reattachment have all been observed. The porosity decreases monotonically with sintering time and scales with the initial porosity and diffusivity along the grain boundary. Deviations from equilibrium pore shapes under slow surface diffusion or fast grain-boundary diffusion conditions yield slower than expected sintering rates.

2.10 Computer Simulation of Final Stage Sintering II: Influence of Initial Pore Size

A two-dimensional Monte-Carlo simulation procedure has been employed to investigate the effect of the initial pore size on the microstructural evolution and the kinetics of final stage sintering. The sintering time scales with r_0^4/D_{gb} and the grain growth time scales with r_0^2/D_m . Pores are found to effectively pin the grain boundaries from the beginning of final stage sintering at a porosity of $\Phi = 0.09$ until $\Phi \leq 0.03$. For $\Phi \leq 0.03$, the remaining pores do not effectively retard grain boundary migration and normal grain growth occurs. Small pores were found to be less effective at retarding grain growth than expected on the basis of a simple grain growth pinning model. The mean pore size was found to be nearly constant throughout the simulations.

2.11 Development of Superplastic Structural Ceramics

Superplastic structural ceramics, Y-TZP, Al_2O_3 , Si_3N_4 and their composites, that can withstand biaxial stretching to large strains, have been developed recently. Microstructural design of these ceramics first calls for an ultrafine grain size that is stable against coarsening during sintering and deformation. A low sintering temperature is a necessary, but not sufficient, condition for achieving the above. In many cases, the selection of an appropriate phase, such as tetragonal phase in zirconia or α phase in silicon nitride which is resistant to grain growth, is crucial. The use of sintering aids and grain growth inhibitors, particularly those that segregate to the grain boundaries, can be beneficial. Second phase particles are especially effective in suppressing static and dynamic grain growth. Another major concern is to maintain an adequate grain boundary cohesive strength, relative to the flow stress, to mitigate cavitation or grain boundary cracking during large strain deformation. Existing evidence suggests that a lower grain boundary energy is instrumental in achieving this objective. The selection of an appropriate phase and the tailoring of the grain boundary or liquid phase composition can sometimes drastically alter the cavitation resistance. Related observations on forming methods, forming characteristics, and sheet formability are also reviewed. It is found that the basic deformation characteristics are akin to diffusional creep and dominated by grain boundary diffusion. However, they are frequently altered by interface reactions,

second-phase hardening/softening, and dynamic grain growth induced strain hardening. Ductility and formability, on the other hand, are controlled by the flow stress and flaw distribution, not by deformation instability as in superplastic metals. Analytical models and empirical correlations are presented to describe various constitutive relations in the above areas.

2.12 Superplastic Flow of Two-Phase Ceramics Containing Rigid Inclusions—Zirconia/Mullite Composites

A continuum theory for non-Newtonian flow of a two-phase composite containing rigid inclusions is presented. It predicts flow suppression by a factor of $(1-V)^q$, where V is the volume fraction of the rigid inclusion and q depends on the stress exponent and the inclusion shape. Stress concentrations in the rigid inclusion have also been evaluated. As the stress exponent increases, flow suppression is more pronounced even though stress concentration is less severe. To test this theory, superplastic flow of zirconia/mullite composites, in which zirconia is a soft, non-Newtonian superplastic matrix and mullite is a rigid phase of various size, shape and amount, is studied. The continuum theory is found to describe the two-phase superplastic flow reasonably well.

III. PROPOSED RESEARCH

3.1 Materials

CeO_2 and Y_2O_3 systems are the focus of the proposal. CeO_2 is a cubic fluorite-structured oxide which has a broad range of nonstoichiometry due to $\text{Ce}^{3+}/\text{Ce}^{4+}$ conversion. The latter feature is well-characterized by several investigators, and it also gives rise to electronic conduction in addition to ionic conduction.[30-33] Solid solutions form between CeO_2 and oxides of divalent Mg, Ca, Sr, trivalent Sc, Yb, Y, Gd, La, tetravalent Ti and Zr, and to a more limited extent pentavalent Nb and Ta, over at least several mole percent. (In the above, the cations within each group are ranked by their ionic radii from small to large ones.) Thus, this system is well-suited for the present study. Y_2O_3 is a cubic oxide of the C type R_2O_3 (R = rare earth element) structure which can be regarded as a derivative structure of anion-deficient fluorite. Its nonstoichiometry is again

attributed to oxygen vacancies and interstitials and allows a considerable range of solid solutions with all of the above cations.^[34] Pure CeO₂, pure Y₂O₃, and their solid solutions with all of the above dopants will be studied in this work.

Powders of CeO₂ will be prepared following the method reported by us. (See Sec. 2.1 and Appendix.) In this method, CeO₂ was found to form directly from an aqueous solution of nitrate with hexamethylenetetramine as the ligand source and oxidizer. The resultant powders are highly sinterable; fully dense ceramics can be obtained easily at 1200°C using die-pressed green bodies. Doping will be achieved by dissolving a salt of the cation dopant in the suspension which carries the undoped CeO₂ powders. The suspension will be gelled and then dried and calcined to obtain a solid solution. Powders of Y₂O₃ will be prepared following the method of Sordelet and Akine,^[6] which uses urea as the ligand source, and doped similarly. These methods have been routinely practiced in our laboratory and work well for the above system.

Although the above description is limited to CeO₂ and Y₂O₃, in the course of the proposed research appropriate attention will also be made to the closely related ZrO₂ system. Zirconia in the tetragonal phase field has been extensively studied by us using a 12mol% CeO₂ stabilized alloy.^[7] The result of this study is entirely consistent from the viewpoint of space charge theory of solute drag. However, the issue of a much higher grain boundary mobility in the cubic phase is still unresolved.^[8] In addition, in the concentrated solid solution, zirconia forms many ordered phases: MZr₆O₁₃, MZr₄O₉, M₆Zr₁₉O₄₆, M₂Zr₅O₁₇, and MZrO₃ for divalent cations, M₂Zr₇O₁₇, M₂Zr₅O₁₃, M₂Zr₂O₇, and M₄Zr₃O₁₂ for trivalent cations, and MZr₃O₈ and MZrO₄ for tetravalent cations. The stability and structures of these compounds vary depending on the size of the M ions. These phases form either on a macroscopic or a microscopic scale, although the temperature regime for the ordering is somewhat limited at times and its kinetics can be quite sluggish.^[35] These aspects will be borne in mind in our research and, when possible, additional parallel experiments will be planned to explore the analogy and differences between these ZrO₂ based ceramics and our focus materials (CeO₂ and Y₂O₃).

3.2 Solute Drag

The central task area of the proposed research is in static grain growth, dynamic grain growth, and related mechanical phenomena which involve processes expected to be influenced by solute drag. Using fully dense specimens, static grain growth experiments and measurements of the average grain boundary mobility will be performed over a wide range of temperatures. They will be complemented by compression tests at several constant strain rates using specimens of fine initial grain sizes to determine the threshold of dynamic grain growth in the diffusional creep regime. Additional deformation experiments using coarse grain materials will also be performed to investigate the solute effect on dislocation creep.

The grain boundary mobility will be analyzed using the model recently developed by us. (See Section 2.3 and Appendices.) A systematic comparison of the mobilities of CeO_2 and Y_2O_3 , with and without various amounts of cation solutes of different charges and sizes, will be made to discern the basic trend. The plausible mechanisms for solute drag and the necessary solute defect interactions will be inferred from such reasoning. The latter considerations are expected to be important at least for CeO_2 . It is known that in CeO_2 a large concentration of Ce^{3+} is present. Therefore, it is relatively easy to maintain within the intrinsic regime and to investigate compensation/competition effects of other solutes within the same regime.^[30-32] In addition, there is now strong evidence to suggest a widely varying degree of solute-oxygen vacancy association which is sensitive to size and charge misfit that can even alter the coordination number of competing cations.^[21-22,25,28] These interactions are expected to have some effect on segregation and diffusion of the solutes. Special attention will be paid to the temperature dependence of grain boundary mobility which is expected to primarily reflect that of solute diffusion in the lattice with some secondary contribution from segregation energy. Other possible phenomena which might be encountered in the data analysis include saturation of the segregant and the grain boundary charge. A model which incorporates all the above considerations will be constructed and the parameters for various contributions will be determined by best fitting the model with the extensive data base obtained in the proposed study. To complete this exercise, we will expect the model to provide

specific new predictions which may be experimentally checked to validate the model. For example, some compensating or competing dopant experiments may be envisioned for this purpose.

Concerning dynamic grain growth, our previous investigation of the Ce-TZP system has established a one-to-one relationship between the static grain growth rate and the dynamic grain growth rate.^[36] In the case of diffusional creep, this correspondence is further extended to the stress-strain curves which manifest varying degrees of work-hardening due to grain size increase.^[36] We will seek to extend similar observations to the CeO₂ and Y₂O₃ systems. A constitutive equation will be constructed to describe the stress-strain curve by incorporating grain growth. At a more fundamental level, it is recognized that the currently available models on dynamic grain growth^[37] do not seem to lend themselves to an immediate explanation of the above relationship between static and dynamic grain growth. Therefore, further microscopic models will be considered in our work to elucidate this point. Lastly, the solute drag model with space charge segregation will be extended to the high velocity regime to examine the consequence of solute breakaway from a moving boundary. The latter task will probably be handled numerically since it is now known that the space charge potential is strongly dependent on the velocity. (See Section 2.3 and Appendix.)

An integral part of the above analysis will consist of microstructural and microchemical characterization at the TEM level. The spatial distributions of solutes near grain boundaries will be selectively established by STEM in order to confirm solute segregation, the designation of grain boundary charge, the space charge compensation by counter ions, and the effect of co-dopants. In the case of dynamic grain growth, selective solute distributions within the grain and on both sides of the grain boundary will have to be measured to reveal evidence of solute breakaway. In the case of deformation, dislocation substructures will be compared at various dopant and concentration levels and grain sizes to discern the effect of aliovalent solutes as very little is currently known of this aspect for solid solution oxides. (Major changes in dislocation substructures and creep mechanisms can be caused by solutes which lower dislocation mobilities, as in some metals.) Lastly, in favorable situations, attempts will also be made to follow kinetics of dislocation loop

shrinkage which should provide data of lattice diffusivity over a very short distance of the order of loop radius.^[38]

3.3 Densification and Coarsening

The focus of this task area is to evaluate the densification and coarsening kinetics using powders conditioned to have a precisely known grain boundary mobility (see Sec. 3.2). Part of the present task will be performed routinely during the screening experiments on grain growth which may find dopants that have unusual effects on sintering and yet behave normally as far as their effects on grain boundary mobility are concerned. Microscopic examination of the intermediate and final microstructures will probably reveal the likely cause of the anomalous effect, for example, by virtue of an unusually large dihedral angle. This information will then allow a more comprehensive assessment of the solute effects on densification and coarsening.

Three sets of experiments are planned in addition to the above. First, we will compare densification kinetics and microstructures of differently doped solid solutions and evaluate these results with the benefit of the knowledge of grain boundary mobility. The results will be analyzed using the established kinetic equations in the literature for sintering and grain growth to (a) separate the contribution of pore drag from solute drag; (b) assess the validity of the pore drag theory given the data of pore shrinkage; and (c) assess the validity of densification-coarsening equation knowing (a) and (b). Such an exercise is necessary for the basic understanding of final stage sintering theory but has not been done definitively to our knowledge, mainly due to the lack of knowledge of the mobility of a pore-free grain boundary.

The second experiment planned is intended to assess the effect of solute drag on the sintering of second phase ceramics. According to the current understanding,^[39-40] the main reason for the sintering retardation by a non-sintering second phase, the so-called constraint effect, is the rapid coarsening of the matrix which increases the matrix creep resistance to render accommodation difficult. This is supported by the observation that viscous sintering of glasses, which is not subject to grain coarsening, is free of the constraint effect.^[40] If so, the sinterability of two-phase ceramics

should also scale inversely with grain boundary mobility. To examine this possibility, we will compare densification kinetics and microstructures of differently doped solid solutions with a second phase. By using the highly sinterable CeO_2 powders, we will be able to perform these experiments at temperatures no higher than 1300°C . This would allow us to use either alumina or SiC as an inert second phase without too much complication due to the change of grain boundary chemistry or second phase coarsening. Microstructural examination will provide additional direct information on the mechanism. Its volume fraction will be kept low enough to avoid the complication of forming a rigid, connected network of the second phase.

The last experiment planned will investigate the effect of initial porosity on sintering of otherwise highly sinterable powders with various grain boundary mobility. We are motivated by our observation in CeO_2 which showed that, even with a green density as low as 25%, fully dense bodies can still be obtained after firing with essentially identical microstructures of only slightly different grain size distributions. This seems to contradict the commonly held notion that claims a direct correspondence between packing density and sinterability.^[41-43] Instead, it would suggest that, if agglomeration can be avoided, no microstructural coarsening happens until final stage sintering, and the transition to the latter occurs over a relatively narrow density regime which is independent of initial porosity.^[44-46] We intend to investigate the above aspect in more detail by conducting sintering experiments using isostatically pressed pellets of different green densities using CeO_2 powders and Y_2O_3 powders, and to repeat such experiments, if necessary, with doped powders. (The use of isostatic pressing rather than colloidal packing techniques is intentional to demonstrate that handling of dried powders does not necessarily introduce agglomeration, as often claimed in the literature, if highly sinterable powders are available, which is the case with CeO_2 .) Microstructural examination to identify the onset of final stage sintering and the final microstructure, including the grain size distribution, will be performed to complement the analysis of densification data.

It should be mentioned that in the past, we have, in collaboration with Professor David J. Srolovitz's group in our department, developed a method to simulate sintering using

Monte-Carlo techniques in two-dimensional triangular lattice.^[46-47] (See also Section 2.) These techniques can be further developed to three dimensions and can certainly be easily modified to incorporate the presence of second phase particles. (Grain boundary mobility is already an independent variable in the current code.) Therefore, given enough resources, we are indeed well-positioned to carry out a highly sophisticated simulation effort in this area to augment the essentially experimental approach outlined above.

3.4 Grain Boundary Electrical Behavior

The objective of this task area is to explore the possible effects of space charged induced solute segregation on grain boundary electrical behavior. The space charge layer can be regarded as providing a large capacitative contribution to the grain boundary electrical component.^[48] The magnitude of this capacitance is dependent on the thickness of the double layer and the total charge stored within. It is unique in that it has no equivalent effect in the bulk. The resistive contribution of the grain boundary component is also expected to be affected by the solute due to the different interactions between solute and oxygen vacancies. The equivalent solute effect on resistance in the bulk, however, is already well-documented in CeO₂ and ZrO₂ and can be neatly correlated to lattice misfit of the solute oxide.^[21,25,49] Systematic and focussed investigations on the grain boundary effect is rare in the literature, other than some reports on impurities (typically SiO₂ and Al₂O₃) which blocked, and sometimes cleansed, the grain boundaries.^[50-53]

To investigate the above behavior, we will use AC impedance spectroscopy as the primary tool. Electrical conductivity of Y₂O₃ and CeO₂ is primarily due to ionic conduction by an oxygen vacancy mechanism, although electronic contribution by a small polaron mechanism is also important in CeO₂ when a significant concentration of Ce³⁺ is present due to reduction or donor doping.^[32] These contributions can be separated from each other by employing the blocked electrode technique. Impedance measurements over a sufficiently wide frequency range can then be used to evaluate grain boundary and bulk conductivity and their results can be analyzed to determine the grain boundary resistance and capacitance. The resistance can usually be analyzed in

a relatively straight-forward manner in terms of carrier concentration and carrier mobility.^[54] Further decoupling of the capacitance into charge and thickness, however, is difficult since only their ratio is measured. Nevertheless, when a sufficiently large data base and the resistance information is available, it may be possible to unambiguously compare these data with the prediction of a segregation model.

The samples used for the above experiments will be prepared in the first task area (Sec. 3.2). They should cover a wide range of grain size, temperature, and solute type and concentration. Among these specimens, a strong segregation effect on grain boundary capacitance is expected for all the grain growth specimens. The actual value of grain boundary capacitance and resistance, however, could be sensitive to the cooling schedule because of the solute redistribution. On the other hand, specimens which have undergone dynamic grain growth and have been quenched afterwards may not show such an effect if solute breakaway has indeed taken place. The grain boundary effect can be progressively brought back by annealing at intermediate temperatures rendering solute diffusion possible. These aspects will be taken into account in designing and interpreting the proposed experiment. Special attention will also be paid to ascertain the influence of grain boundary glassy phase, and, if needed, to use specially prepared specimens free of Si to avoid the potential complication caused by this impurity.^[51]

IV. REFERENCES

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V. EQUIPMENT AND FACILITIES

5.1 The Ceramic Laboratory

Excellent equipment and facilities currently exist in the laboratories of the ceramic group under Professor Chen. They provide almost all the essential processing capabilities for oxides, nitrides and carbides and offer outstanding analytical and mechanical testing facilities especially suited for structural ceramic research. A large part of the equipment and facilities were purchased during the last two and half years, with funds from several major equipment grants from DOE, NSF and AFOSR totaling \$800,000. In addition, the University has cost-shared a total of \$200,000 and has provided Professor Chen \$300,000 when he joined the Department in 1986. Major items are listed below:

A. Furnaces and Hot Press

- Centorr M60 High Vacuum Physical Testing Furnace (2200°C) with tungsten mesh heating elements, physical test kit, hot rods and dies for tension, compression, and extrusion;
- Astro HP-4560-FP20 Hot Press (2200°C) with graphite heating elements for vacuum and inert gases, Honeywell programmable temperature control;
- Centorr EP Elevated Pressure Furnace (2200°C) with graphite heating elements, operating pressure up to 1500 PSIG in inert gases;
- Astro 1000-4560 Graphite Furnace (2200°C) for vacuum and inert gases with programmable temperature control;
- Several super-Kanthal furnaces (1700°C), two platinum furnaces (1500°C), assorted furnaces for lower temperatures;
- Several pyrometers, including an Ircon 7000 infrared radiation thermometer with electrical output for closed-loop control;
- Vacuum Tube Industry KC-MC Dual Frequency Induction Generator (20 kw); and
- IPS Hot Isostatic Press (2000°C) with a graphite, a molybdenum, and a Ni-Cr furnace for pressures up to 200 MPa.

B. Analytical and Powder Preparation

- Fully computerized Rigaku rotating anode X-ray spectrometer with high temperature (1300°C) and cryogenic temperature (77°K) chamber, pole figure goniometer;
- Fully computerized Netzsch STA 409 Simultaneous TG-DTA (1600°C);
- HP 4192A LF Impedance Analyzer (5Hz to 13MHZ);
- Micromeritics 5000ET Sedigraph particle size analyzer;
- Micromeritics Model 9310 Mercury Intrusion Pore Sizer with computerized data acquisition system;
- Yamato Model GA-21 Spray Dryer;
- Theta Model 1600C Dilatron dilatometer;

- Micromeritics 2100 surface area analyzer;
- Brookfield Cone-Plate Viscometer
- Autoclave Engineering MAWP Isostatic Press (410 MPa);
- Harig 612 Autostep Surface Grinder with numerical control; and
- Assorted powder preparation attrition mills, ball mills, homogenizers, centrifuge, analytical and wet chemical instruments.

C. Mechanical Testing

- Two fully instrumented and computer-controlled MTS 810 Servohydraulic Testing Machines with hydraulic grips, high temperature extensometers, several high resolution extensometers with 16 bit high resolution data acquisition systems;
- One fully instrumented and computer-controlled Instron Model 1335 Servohydraulic Test Machine mounted with a high pressure (800 MPa) vessel, triaxial test fixtures, internal load cells and wire-wound furnaces, fluid and gas pressurization capabilities up to 1400 MPa;
- One fully instrumented, computer-controlled, electromechanically actuated Instron Model 1362 Test Machine with fixtures for tension, compression, three-point and four-point bending, and biaxial sheet-forming for ceramics up to 1450°C, swing-arm mounted with two physical testing furnaces with platinum and molybdenum heating elements; and
- Zwick Microhardness Tester; and
- Assorted electronics, oscilloscopes, and meters for electrical, acoustic and gamma-ray measurements.

5.2 Department of Materials Science and Engineering and Electron Microscopy Analysis Laboratory (EMAL)

Excellent analytical and characterization facilities, including three state-of-the-art electron microscopes purchased in the last two years, are available in the Department and in the University laboratory EMAL. Ceramic students and staff have been major users of these facilities which are

located in close vicinity to the ceramic laboratories. A partial list of major equipment relevant to the present research is given below:

- JEOL JEM-2000FX analytical scanning electron microscope equipped with Tracor-Northern high angle (30 mm²) and horizontal light element thin window EDS detectors (30 mm²), Gatan parallel EELS, and Tracor-Northern 5500 data analysis systems;
- JEOL JEM-4000EX high resolution electron microscope equipped with a Gatan 622 JAG image intensifier system with capabilities of 0.17 nm point-to-point resolution;
- JEOL JEM-100CX analytical STEM, with capabilities for high resolution (0.2 nm line) imaging, SAD, EDS, CBED, EELS, electron channeling;
- Two Hitachi Models S520 and S800 microprocessor-based SEM's with XEDS, computer controlled with signal digitizers, low voltage imaging capabilities (500V);
- CAMECA MBX electron microprobe analyzer with KEVEX 8000 EDS;
- TEM specimen preparation facilities: Technics, Inc. micro-ion milling unit, Struers Tenupol thinning apparatus and cut-off machine, Southbay Technologies backthinning apparatus, and VCR D-500 dimpler;
- Perkin Elmer Phi 5400 series ESCA/XPS;
- Fully instrumented and computerized Perkin-Elmer FTIR;
- Nikos hot-hardness tester; and
- All necessary optical microscopy facilities.

VI. PERSONNEL

The program will be directed by Professor I-Wei Chen. He will be assisted by three graduate students. Professor Chen has extensive expertise in materials research. In recent years, he has been mostly interested in structural ceramics and has devoted his attention to developing a comprehensive approach which integrates crystal chemistry, alloy design, advanced powder

processing, microstructure and property characterization, and mechanistic modelling to explore novel ceramics. The research proposed here is in line with his current interest and thrust of research. He will be assisted by three full-time graduate students who will be working on (a) grain growth experiments and powder development; (b) sintering studies and modeling/computer simulation; and (c) AC impedance spectroscopy investigation of grain boundary electrical behavior and STEM analysis. Their research in these areas is expected to lead to three Ph.D. theses.

Partial support (20%) is also requested for a secretary to work full time for Professor Chen. She will be responsible for assisting Professor Chen on managing the program and on preparation of reports and publications.

VII. PREVIOUS RELATED WORK OF THE PRINCIPAL INVESTIGATOR

The following papers, which have been presented by Professor Chen and his coworkers in the Annual Meetings of the American Ceramic Society during the last two years, are representative of their recent work in ceramics. Abstracts of the presentations were published in the conference abstract books in 1991 and 1992.

A. Papers Presented at the 93rd Annual Meeting of The American Ceramic Society (May 1991):

1. "X-ray Absorption Studies of ZrO_2 with Trivalent Dopants," P. Li, I-W. Chen, J.E. Penner-Hahn and T.Y. Tien.
2. "Alumina-Aluminate Platelet Composite Materials," P-L. Chen and I-W. Chen.
3. "Crack-Tip Fields for Transformation Toughened Materials," J. Pan and I-W. Chen.
4. "Solute Effect on the Grain Growth of CeO_2 ," L.A. Xue and I-W. Chen.
5. "Control of Grain Boundary Pinning in Al_2O_3/ZrO_2 Composites with Ce^{3+}/Ce^{4+} Doping," L.A. Xue, K. Meyer, and I-W. Chen.
6. "Shear-Thickening Creep in Glass Containing Silicon Nitrides," S-L. Hwang and I-W. Chen.
7. "Transient-Liquid Aided Superplastic Forming of Silicon Nitrides," S-L. Hwang and I-W. Chen.

8. "Formability of Superplastic Si_3N_4 in Uniaxial and Biaxial Deformation," X. Wu and I-W. Chen.
9. "Superplastic Mullite Via Transient Phase Processing," L.A. Xue and I-W. Chen.
10. "Uniaxial Tension-Compression Cyclic Fatigue of Alumina," L.J. Malczewski and I-W. Chen.
11. "Cyclic Fatigue of Yttria-Stabilized Zirconia and Magnesia-Stabilized Zirconia," S-Y. Liu and I-W. Chen.
12. "The Role of R-Curve and K-a Curve on the Fatigue Fractography of Monolithic Ceramics," S-Y. Liu and I-W. Chen.

B. Papers Presented in the 94th Annual Meeting of The American Ceramic Society (April 1992):

1. "Reactive CeO_2 Powders by the Homogeneous Precipitation Method," P.L. Chen and I-W. Chen.
2. "Solute Effect on Grain Growth in Y_2O_3 and CeO_2 Systems," P.L. Chen and I-W. Chen.
3. "A New SiC_w Reinforced Lithium Aluminosilicate Composite," L.A. Xue and I-W. Chen.
4. "Superplastic Alumina at Temperature Below 1300°C," L.A. Xue and I-W. Chen.
5. "X-Ray Absorption Studies of PMN-PZN Relaxors," P.Li, S.Y. Chen and I-W. Chen.
6. "The Effects of Alumina Content on the Transformation Plasticity and R-Curve Behaviors of Ce-TZP/ Al_2O_3 Composites," J.S. Cherng and I-W. Chen.
7. "Reactive Sintering Characteristics of Si_3N_4 with Liquid Trapping Inclusions," S.L. Hwang and I-W. Chen.
8. "Microstructures of Superplastic Silicon Nitrides," S.L. Hwang and I-W. Chen.
9. "Plasticity Induced Fatigue Damage in Ce-TZP," S.Y. Liu and I-W. Chen.
10. "Environmental and Mechanical Contributions to Cyclic and Static Fatigue in Si_3N_4 and Al_2O_3 ," D. Jacobs and I-W. Chen.
11. "Crack Propagation of a High Strength Si_3N_4 at 1400°C Under Creep and Fatigue Conditions," X. Wu, T-S. Sheu, T.Y. Tien and I-W. Chen.
12. "Crystal Chemistry Studies of ZrO_2 Solid Solutions by EXAFS," P. Li, J.E. Penner-Hahn, T.Y. Tien and I-W. Chen.
13. "Deformation and Fracture of Two-Phase Ceramics at Elevated Temperatures," I-W. Chen.
14. "XAS: A Probe to Local Atomic and Electronic Structures of Ceramics," P. Li and I-W. Chen.

VIII. RESUME OF THE PRINCIPAL INVESTIGATOR

I-WEI CHEN

Position	Professor (1990-present) Department of Materials Science & Engineering University of Michigan, Ann Arbor Tel: 313-763-6661 Fax: 313-763-4788
Education	Ph.D. Metallurgy, Massachusetts Institute of Technology (1980) Thesis: "Creep Cavitation in Type 304 Stainless Steel" M.S. Physics, University of Pennsylvania, Philadelphia B.S. Physics, National Tsinghua University, Taiwan
Employment	Associate Professor (1986-1990) Department of Materials Science & Engineering University of Michigan Assistant Professor (1980-1986) Department of Nuclear Engineering & Department of Materials Science and Engineering Massachusetts Institute of Technology
Scientific Societies	Fellow of The American Ceramic Society Member of The Ceramic Society of Japan, The American Physical Society and The Metallurgical Society of AIME

I-Wei Chen is best known for his work on phase transformation and mechanical properties of ceramics and metals. His thesis on stainless steel under Professor A.S. Argon and the subsequent research have been cited often in the literature of creep and creep fracture. Since 1983 he has reported a series of *in-situ* HREM small particle experiments on zirconia, which established the mechanisms for nucleation and growth of martensitic transformation. He also introduced high pressure testing techniques to studies of zirconia ceramics; such work has constituted much of the current basic understanding of zirconia transformation plasticity. At the University of Michigan, his group has been investigating processing and properties of brittle solids broadly, including fracture, fatigue, sintering, grain growth and atomic structure. It has also developed a series of ultrafine-grained ceramics which exhibit remarkable superplastic behavior (zirconia, alumina, mullite, silicon nitride and their composites). In addition to his experimental work, Professor Chen is interested in theoretical and computer modelling of complex phenomena in micromechanics, kinetics and microstructure. He served for The American Ceramic Society as Program Chair of the Basic Science Section during 1989-1990 and is currently an Associate Editor of the *Journal of the American Ceramic Society*.

Courses Taught (since July 1986)

1. Undergraduate Courses

- MSE 250: Principles of Engineering Materials
- MSE 350: Structure and Properties of Materials (with W.F. Hosford and R.E. Robertson)
- MSE 440: Ceramic Materials

2. Graduate Courses

- MSE 552: Reactions in Ceramic Processes
- MSE 553: Structures of Ceramic Compounds
- MSE 554: Properties of Ceramic Compounds
- MSE 574: High Temperature Materials
- MSE 620: Phase Transformations in Solids

Ph.D. Thesis Supervised

1. "Nucleation and Growth Processes of Martensitic Transformation in ZrO_2 Particles," Y.H. Chiao, 1986.
2. "An Experimental Study of Constitutive Relations of Transformation Plasticity in Zirconia Based Ceramics," P.E. Reyes-Morel, 1986.
3. "Superplastic Flow of Mullite-Zirconia Composites," C.K. Yoon, 1990.
4. "Mixed Valence Niobium (IV,V) Perovskite Solid Solutions and Phase Relations, Crystal Chemistry, Electrical and Magnetic Properties," S.J. Keating, (co-chaired with T.Y. Tien) 1992.
5. "Deformation Processing of Ceramics," X. Wu, 1991.
6. "Fatigue of Zirconia Ceramics," S-Y. Liu, 1992.
7. "Transformation Toughening in Ce-stabilized Zirconia," J.S. Cherng, 1992.
8. "Superplasticity and Shear Thickening in Sialon Ceramics," S.L. Hwang, expected in 1992.
9. "Near Edge and Extended X-ray Absorption Fine Structure Studies of Ceria and Zirconia Compounds," P. Li, expected in 1992 (co-chaired with T.Y. Tien).

M.S. Thesis Supervised

1. "Magnetic Effect on Phase Transformation in Fe-Cr Alloys," E. Faillace, 1984.
2. "Creep Cavitation in Astrolloy," M. Capano, 1984 (co-chaired with A. S. Argon).
3. "The Effect of Solute Segregation on Void Nucleation," A. Taiwo, 1985.
4. "Deformation of Magnesia-Partially-Stabilized Zirconia Under Triaxial Compression," P.E. Reyes-Morel, 1985.
5. "Fatigue Crack Propagation in Silicon Nitride," D. Jacobs, 1991.
6. "Ceramic-Metal Composites Manufactured by Melt Infiltration," M.Y. Yoon, 1991.
7. "Alumina-Platelet Aluminate Composites," P.L. Chen, 1991.
8. "Uniaxial Tension-Compression Cyclic Fatigue of Alumina," L.J. Malczewski, 1991.
9. "Drying Cracks in $BaTiO_3$ Thin Film from Metal Organic Solutions," S.Y. Chen, expected, 1992.

Publications

1. I-W. Chen and A.S. Argon, "Grain-boundary and Interface Boundary Sliding in Power Law Creep," *Acta Metallurgica*, 27, 749-754 (1979).
2. I-W. Chen and A.S. Argon, "Steady State Power Law Creep in Heterogeneous Alloy with Coarse Microstructures," *Acta Metallurgica*, 27, 785-791 (1979).

3. A.S. Argon, I-W. Chen and C.W. Lau, "Intergranular Cavitation in Creep," in Creep Fatigue Interactions, Eds. R.M.N. Pelloux and N.S. Stoloff, AIME, 46-85 (1979).
4. A.S. Argon, I-W. Chen and C.W. Lau, "Mechanics and Mechanisms of Intergranular Cavitation in Creeping Alloys," in Three Dimensional Constitutive Relations and Ductile Fracture, Ed. S. Nemat-Nasser, North Holland Pub., 23-49 (1980).
5. M.P. Cleary, I-W. Chen and S-M. Lee, "Self-consistent Techniques for Heterogeneous Media," *J. Engineering Mechanics, ASCE*, 106, 861-887 (1980).
6. I-W. Chen and A.S. Argon, "Creep Cavitation in 304 Stainless Steel," *Acta Metallurgica*, 29, 1321-1333 (1981).
7. I-W. Chen and A.S. Argon, "Diffusive Growth of Grain-boundary Cavities," *Acta Metallurgica*, 29, 1759-1768 (1981).
8. I-W. Chen and A.S. Argon, "Nucleation and Growth of Intergranular Cavities During Creep of Type 304 Stainless Steel," in Creep and Fracture of Engineering Materials, Eds. B. Wilshire and D.R.J. Owen, Pinerage Press, 289-302 (1981).
9. I-W. Chen, "Migration-assisted Diffusional Creep by Grain-boundary Diffusion," *Acta Metallurgica*, 30, 1317-1323 (1982).
10. I-W. Chen, "Diffusional Creep in Two-Phase Materials," *Acta Metallurgica*, 30, 1655-1664 (1982).
11. I-W. Chen, "Mechanisms of Cavity Growth in Creep," *Scripta Metallurgica*, 17, 17-22 (1983).
12. I-W. Chen, "Irradiation Induced Segregation in Concentrated Alloys," *J. Nuclear Materials*, 116, 249-259 (1983).
13. I-W. Chen and Y-H. Chiao, "Martensitic Nucleation in ZrO₂," *Acta Metallurgica*, 31, 1627-1638 (1983).
14. I-W. Chen, "Cavity Growth on a Sliding Grain Boundary," *Metallurgical Transactions*, 14A, 2289-2293 (1983).
15. I-W. Chen, "Effects of Boundary Mobility and Phase Equilibrium on Kinetic Processes of Multi-component Polyphase Ceramics," in Advances in Ceramics, Eds. M.F. Yan and A.H. Heuer, American Ceramic Society, 6, 224-235 (1983).
16. I-W. Chen, E. Faillace and A.P. Miodownik, "The Effect of a Magnetic Field on Phase Transformations in FeCr Alloys," in Ferritic Steels in Nuclear Technologies, Eds. J.W. Davis and D.J. Michel, AIME, 517-524 (1984).
17. I-W. Chen, "Magnetic Field Induced Growth of Ferromagnetic Materials Under Irradiation," in Ferritic Steels in Nuclear Technologies, Eds. J.W. Davis and D.J. Michel, AIME, 525-531 (1984).
18. I-W. Chen and Y-H. Chiao, "Martensitic Nucleation in ZrO₂ and HfO₂ - An Assessment of Small Particle Experiments with Metal and Ceramic Matrices," in Advances in Ceramics, Eds. N. Claussen, M. Rühle and A.H. Heuer, American Ceramic Society, 12, 33-45 (1984).

19. I-W. Chen, "Anisotropic Diffusion of Point Defects to an Edge Dislocation," *J. Nuclear Materials*, **125**, 52-63 (1984).
20. I-W. Chen and M.H. Yoo, "Creep Cavitation under Interface Segregation," *Acta Metallurgica*, **32**, 1499-1508 (1984).
21. I-W. Chen, "Irradiation Growth Due to Magnetodiffusion," *J. Nuclear Materials*, **133 & 134**, 435-438 (1985).
22. E. Faillace and I-W. Chen, "The Effects of a Strong Magnetic Field on Age-hardening of Iron-chromium Alloys," *J. Nuclear Materials*, **133 & 134**, 343-346 (1985).
23. I-W. Chen and Y-H. Chiao, "Theory and Experiment of Martensitic Nucleation in ZrO_2 -containing Ceramics and Ferrous Alloys," *Acta Metallurgica*, **33**, 1827-1845 (1985).
24. I-W. Chen, Y-H. Chiao and K. Tsuzaki, "Statistics of Martensitic Nucleation," *Acta Metallurgica*, **33**, 1847-1859 (1985).
25. I-W. Chen and Ademola Taiwo, "Nucleation of Voids--The Impurity Effect," in Proceedings of the Twelfth International Symposium on the Effects of Radiation on Materials, Eds. F.A. Garner and J.S. Perrin, American Society Testing and Materials, *ASTM-STP 870*, 507-524 (1985).
26. I-W. Chen, "Superplastic Flow of Two-phase Alloys," in Superplasticity, Eds. B. Baudelot and M. Suery, Editions du CNRS, Paris, 5.1-5.20 (1985).
27. I-W. Chen, "Mechanisms of Transformation and Transformation Plasticity in ZrO_2 -containing Ceramics," in Zirconia Ceramics 4, Eds. S. Somiya and M. Yoshimura, Tokyo Institute of Technology, 55-79 (1985).
28. Y-H. Chiao and I-W. Chen, "In-Situ TEM Observations of the Structures and Migration of Martensitic Interface in Small ZrO_2 Particles," in Grain and Boundary Structure and Related Phenomena, Suppl. Trans. Japan Inst. Metals, **27**, 197-203 (1986).
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30. I-W. Chen and Y-H. Chiao, "Martensitic Nucleation in Small ZrO_2 Particles," in Phase Transitions in Condensed Systems: Experiments and Theory, Eds. C.S. Cargill, F. Spaepen and K.N. Tu, Symposium Proceeding Series, **57**, 149-158 (1986).
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62. L.A. Xue and I-W. Chen, "Deformation and Grain Growth of Low Temperature Sintered High Purity Alumina," *J. of American Ceramic Society*, 73 [11] 3518-21 (1990).

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79. X. Wu and I-W. Chen, "Exaggerated Texture and Grain Growth of a Superplastic Silicon Nitride," accepted for publication, *J. of American Ceramic Society* (1991).

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83. L.A. Xue and I-W. Chen, "A New SiC Whisker Reinforced Lithium Aluminosilicate Composite," submitted for publication to *J. of American Ceramic Society* (1992).
84. P.L. Chen and I-W. Chen, "Reactive CeO₂ Powders by Homogeneous Precipitation Method," submitted for publication to *J. of American Ceramic Society* (1992).
85. I-W. Chen, "Solute Drag on Grain Boundary in Ionic Solids—The Space Charge Effect," in Design and Control of Grain Boundary in Ceramics, Eds. R. Ishizaki and K. Nihara, Elsevier Publications, New York (1992).

Invited Talks

1. "Creep Cavitation—An Overview," at National Bureau of Standards, Washington D.C., January, 1981.
2. "Cavity Growth on Grain Boundaries," at Berkeley Nuclear Laboratories, Electric Generating Board, Berkeley, Gloucestershire, U.K., April, 1981.
3. "Elastic and Magnetic Anisotropy of Point Defects—Their Role in Dimensional Instability of Irradiated Metals," at Kernforschungsanlage, Institut fur Festkorperforschung, Julich, Federal Republic of Germany, July, 1983.
4. "Superplasticity in Two Phase Alloys," at Symposium on Large Strain Deformation, TMS-AIME, Philadelphia, PA, October, 1983.
5. "Constitutive Equations of Superplastic Materials," at International Nickel Company, Sterling Forest, NY, December, 1983.
6. "Martensitic Nucleation in Metal Ceramic Composites," at IBM Watson Research Center, Yorktown Heights, NY, February, 1984.
7. "Martensitic Transformations in Ceramics," at Los Alamos National Laboratory, Los Alamos, NM, May, 1984.
8. "Creep Deformation of Multiphase Ceramics," at Gordon Research Conference on Solid State Studies in Ceramics, New London, NH, July, 1984.
9. "Mechanisms of Transformation and Transformation Plasticity in ZrO₂," at Zirconia Ceramics 5, Tokyo Institute of Technology, Yokohama, Japan, December, 1984.
10. "Transformation Plasticity in Brittle Solids," at Division of Applied Sciences, Harvard University, MA, April, 1985.

11. "Martensitic Nucleation in ZrO_2 ," Second International Workshop on Transformation Toughening," Lorne, Australia, April, 1985.
12. "Quasi-static Intergranular Fracture at 0.5 T_m —Mechanisms of Stress Relief Cracking in Low Alloy Steels," at Monash University, Australia, April, 1985.
13. "Superplastic Flow of Two-Phase Alloys," at International Conference on Superplasticity, Grenoble, France, September, 1985.
14. "Implications of Transformation Plasticity on Fracture Control of ZrO_2 Ceramics," at Zirconia Ceramics 7, Tokyo Institute of Technology, Yokohama, Japan, November, 1985.
15. "Displacive Transformation in ZrO_2 ," at Ceramic Science Symposium, Basic Science Section of The American Ceramic Society, Baltimore, MD, November, 1985.
16. "Transformation, Transformation Plasticity and Transformation Toughening in ZrO_2 Containing Ceramics," at University of Pennsylvania, PA, November, 1985.
17. "Transformation, Transformation Plasticity and Transformation Toughening in ZrO_2 Containing Ceramics," at Ohio State University, OH, January, 1986.
18. "A Comparative Study of Transformation Plasticity and Toughening in Mg-PSZ and Ce-TZP," at Advanced Ceramics 2, Tokyo Institute of Technology, Yokohama, Japan, September, 1986.
19. "Interface Effects on the Deformation and Failure of High Temperature Alloys," at TMS Southeast Regional Meeting, Oak Ridge, TN, September, 1986.
20. "Ceramic Steels—The Zirconia Containing Ceramics," at Michigan Chapter of TMS, Southfield, MI, September, 1986.
21. "Transformation, Transformation Plasticity and Transformation Toughening of Zirconia Containing Ceramics," at Symposium on Toughening via Stress-induced Transformation, TMS Fall Meeting, Orlando, FL, October, 1986.
22. "Transformation, Transformation Plasticity and Transformation Toughening of Zirconia Containing Ceramics," at Materials Science Division, Argonne National Laboratory, Argonne, IL, November, 1986.
23. "Transformation Plasticity and Transformation Toughening in PSZ and TZP," at Symposium on Advanced Structural Ceramics, Materials Research Society, Fall Meeting, Boston, MA, December, 1986.
24. "Transformation Plasticity and Transformation Toughening," at Solid Mechanics Division, Brown University, Providence, RI, December, 1986.
25. "A Perspective of Zirconia Research," at Michigan Section of The American Ceramic Society, Detroit, MI, December, 1986.
26. "Transformation and Transformation Plasticity," at Michigan State University, East Lansing, MI, February, 1987.
27. "Transformation, Transformation Plasticity and Transformation Toughening of Zirconia Ceramics," at McMaster University, Canada, March, 1987.

28. "Transformation and Transformation Toughening of Zirconia Ceramics," at Allied-Signal, Morristown, NJ, April, 1987.
29. "A Statistical Theory of Grain Growth," at International Symposium on Progress in Microstructure-1987, Aachen, Federal Republic of Germany, May, 1987.
30. "Sintering and Grain Growth of Tetragonal and Cubic Zirconia," at Sintering '87, Tokyo, Japan, November, 1987.
31. "Texture Development in $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ by Hot Extrusion and Hot Pressing," at Sintering '87, Tokyo, Japan, November, 1987.
32. "Phases, Microstructures and Phase Transformations in Zirconia," at Larry Van Vlack Symposium, 15th Automotive Materials Conference, Ann Arbor, MI, April, 1988.
33. "Advanced Concepts in Toughening of Ceramics," at Weitenburg Workshop on Structural Ceramics, Weitenburg, Federal Republic of Germany, April, 1988.
34. "Crystal Chemistry and Phase Stability of High Tc Superconductors," at the 89th Annual Meeting of The American Ceramic Society, Cincinnati, OH, May, 1988.
35. "Superplasticity of Mullite Zirconia Composites," at MRS International Symposium on Superplastic Forming, Tokyo, Japan, May-June, 1988.
36. "Transformation Toughening," at Gordon Conferences on Solid State Studies of Ceramics, Meriden College, NH, August, 1988.
37. "Superplastic Forming of Ceramics," at US-Japan Workshop on the Processing of Advanced Ceramics, Seattle, WA, August, 1988.
38. "Ceramic Research at The University of Michigan," at Detroit Section of The American Ceramic Society, Detroit, MI, November, 1988.
39. "Recent Progress in Zirconia as a Structural Ceramic," at Department of Materials Science and Engineering, Northwestern University, Evanston, IL, February, 1989.
40. "Structural Ceramic Research at The University of Michigan," at SAMPE Michigan Tri-City Chapter, Midland, MI, February, 1989.
41. "Recent Progress in Zirconia Research," at Department of Materials Science and Engineering, University of Utah, Salt Lake City, UT, July, 1989.
42. "Superplastic Forming of Ceramics," at Gordon Conference on Solid State Studies of Ceramics, Colby Sawyer College, NH, August, 1989.
43. "Superplasticity of Fine Grained Zirconia Polycrystals," at the Fourth International Conference on the Science and Technology of Zirconia, Anaheim, CA, October, 1989.
44. "Transformation Toughening," at ASME Winter Meeting, San Francisco, CA, December, 1989.
45. "Superplastic Forming of Fine-Grained Ceramics," at Department of Materials Science and Engineering, University of Washington, Seattle, WA, January, 1990.

46. "Superplastic Forming of Fine-Grained Ceramics," at the Third International Conference on Ceramic Powder Processing Science, San Diego, CA, February, 1990.
47. "Superplastic Ceramics," at Albrecht-Rabenau Symposium on Contemporary Issues in Ceramic Science, Tegernsee, Germany, July 1990.
48. "Superplastic Forming of Ceramic Composites," at Symposium on Composites, Orlando, FL, November, 1990.
49. "Solute Drag and Grain Growth," at W.E. Heraeu's Seminar on Modelling of Sintering Processes, Bad Honnef, Germany, November, 1990.
50. "Constitutive Equations of Cyclic Fatigue of Ceramics," at the Engineering Foundation Conference on Fatigue of Advanced Materials, Santa Barbara, CA, January, 1991.
51. "Microstructural Design of Superplastic Ceramics," at the International Conference on Superplasticity of Advanced Materials, Osaka, Japan, June, 1991.
52. "X-ray Absorption Studies of ZrO_2 and CeO_2 ," at the Atomic Structure, Bonding and Properties of Ceramics Meeting, Basic Science Division, The American Ceramic Society, Marco Island, FL, October, 1991.
53. "Deformation Processing of Ceramics," at Purdue University, West Lafayette, IN, November, 1991.
54. "Tailoring of Grain Boundary and Bulk Properties by Grain Boundary Alloying," at Japan Fine Ceramic Center Workshop on Materials Processing and Design Through Better Control of Grain Boundaries, Nagoya, Japan, March, 1992.
55. "Fatigue of Tough Ceramics," in 15th Michigan Ceramic Materials Conference on Tough Ceramics, Ann Arbor, MI, March, 1992.
56. "XAS: A Probe to Local Atomic and Electronic Structures of Ceramics," at the Annual Meeting of The American Ceramic Society, Minneapolis, MN, April, 1992.
57. "Deformation and Fracture of Two-Phase Ceramics at Elevated Temperatures," at the Annual Meeting of The American Ceramic Society, Minneapolis, MN, April, 1992.
58. "Transformation, Transformation Plasticity and Transformation Toughening in Ce-TZP/ Al_2O_3 Composites," at the Fifth International Conference on the Science and Technology of Zirconia, Melbourne, Australia, August, 1992.

Other Recent Presentations (since July 1986; * for the presenting author)

1. "Mechanisms and Mechanics of Crack Propagation at 0.5 Tm Under Intergranular Segregation", I-W. Chen,* at Symposium on Crack Propagation Under Creep and Creep Fatigue, American Society for Metals, Orlando, FL, October, 1986.
2. "Reversible Transformation Plasticity in Uniaxial Tension Compression Cycling of Mg-PSZ," K.J. Bowman,* P.E. Reyes-Morel and I-W. Chen, at Symposium on Advanced Structural Ceramics, Materials Research Society Fall Meeting, Boston, MA, December, 1986.

3. "Transformation Plasticity of Zirconia Composites," P.E. Reyes-Morel and I-W. Chen,* at The American Ceramic Society Annual Meeting, Pittsburgh, PA, April, 1987.
4. "Structure and Property of Oxygen-rich $\text{YBa}_2\text{Cu}_3\text{O}_x$," I-W. Chen,* S.J. Keating, X. Wu, J. Hsu, C. Keating and T.Y. Tien, at The American Ceramic Society Annual Meeting, Pittsburgh, PA, April, 1987.
5. "On the Ferroelastic Domain Reorientation Mechanism in Tetragonal Zirconia Polycrystals," B.S. Li, P. Yang, P.E. Reyes-Morel, J.S. Cherng, I-W. Chen* and T.Y. Tien, at The American Ceramic Society Annual Meeting, Pittsburgh, PA, April, 1987.
6. "Shape Memory Effect in Ce-TZP," P.E. Reyes-Morel, J.S. Cherng* and I-W. Chen, at The American Ceramic Society Annual Meeting, Pittsburgh, PA, April, 1987.
7. "Transformation-induced Creep and Creep-fatigue Interactions in Mg-PSZ at Low Temperatures," P.E. Reyes-Morel,* K.J. Bowman and I-W. Chen, at The American Ceramic Society Annual Meeting, Pittsburgh, PA, April, 1987.
8. "Fully Reversed Cyclic Plastic Deformation of Mg-P/SZ," K.J. Bowman,* P.E. Reyes-Morel and I-W. Chen, at The American Ceramic Society Annual Meeting, Pittsburgh, PA, April, 1987.
9. "Defect Structures in Tetragonal, Orthorhombic and Monoclinic Zirconia," Y-H. Chiao* and I-W. Chen, at The American Ceramic Society Annual Meeting, Pittsburgh, PA, April, 1987.
10. "Radiation Effects on Mechanical Properties of Zirconia Based Ceramics", P.E. Reyes-Morel* and I-W. Chen, at the 89th Annual Meeting of The American Ceramic Society, Cincinnati, OH, May, 1988.
11. "Strength and Toughness of Zirconia Ceramics under Triaxial Stress States," J.S. Cherng* and I-W. Chen, at the 89th Annual Meeting of The American Ceramic Society, Cincinnati, OH, May, 1988.
12. "Superplastic Flow in Mullite Zirconia Composites I: The Volume Fraction Effect," C.K. Yoon* and I-W. Chen, at the 89th Annual Meeting of The American Ceramic Society, Cincinnati, OH, May, 1988.
13. "Superplastic Flow in Mullite Zirconia Composites II: The Aspect Ratio Effect," C.K. Yoon* and I-W. Chen, at the 89th Annual Meeting of The American Ceramic Society, Cincinnati, OH, May, 1988.
14. "Grain Growth in Tetragonal and Cubic Zirconia," I.G. Lee* and I-W. Chen, at the 89th Annual Meeting of The American Ceramic Society, Cincinnati, OH, May, 1988.
15. "Deformation Texture of Transformation Toughened Zirconia," K.J. Bowman* and I-W. Chen, at the 89th Annual Meeting of The American Ceramic Society, Cincinnati, OH, May, 1988.
16. "Fatigue and Cyclic Deformation of Mg-PSZ," S-Y. Liu,* K.J. Bowman and I-W. Chen, at the 89th Annual Meeting of The American Ceramic Society, Cincinnati, OH, May, 1988.
17. "Thermal Properties and Processing of Bi-Sr-Ca-Cu-O Superconductors," S.J. Keating,* I-W. Chen and T.Y. Tien, at the 1988 Fall Meeting of Materials Research Society, Boston, MA, November 28-December 3, 1988.

18. "Hot Extrusion of $\text{Bi}_2(\text{Sr,Ca})_3\text{Cu}_2\text{O}_{8+x}$ and $\text{YBa}_2\text{Cu}_3\text{O}_{6+x}$ Wires," X. Wu,* P.E. Reyes-Morel and I-W. Chen, at the 1988 Fall Meeting of Materials Research Society, Boston, MA, November 28-December 3, 1988.
19. "Creep and Hot Extrusion of High Tc Superconductors," X. Wu* and I-W. Chen, at the 91st Annual Meeting of The American Ceramic Society, Indianapolis, IN, April 24-27, 1989.
20. "Oxygen Stoichiometry and Phase Stability in the Sr-Nb-O System," S.J. Keating,* I-W. Chen and T.Y. Tien, at the 91st Annual Meeting of The American Ceramic Society, Indianapolis, IN, April 24-27, 1989.
21. "Monte-Carlo Simulation of Sintering and Grain Growth I: Periodic Microstructure," G. Hassold,* I-W. Chen and D.J. Srolovitz, at the 91st Annual Meeting of The American Ceramic Society, Indianapolis, IN, April 24-27, 1989.
22. "Monte-Carlo Simulation of Sintering and Grain Growth II: Final Stage Sintering," G. Hassold, I-W. Chen* and D.J. Srolovitz, at the 91st Annual Meeting of The American Ceramic Society, Indianapolis, IN, April 24-27, 1989.
23. "Fatigue Life Time Prediction and Fracture Statistics of Brittle Ceramics," S-Y. Liu* and I-W. Chen, at the 91st Annual Meeting of The American Ceramic Society, Indianapolis, IN, April 24-27, 1989.
24. "A Critical Assessment of Crack Tip Plastic Zone in Transformation Toughened ZrO_2 Ceramics," I-W. Chen,* J. Pan, F. Li and J.S. Cherng, at the 91st Annual Meeting of The American Ceramic Society, Indianapolis, IN, April 24-27, 1989.
25. "Superplasticity in Y_2O_3 -stabilized Cubic ZrO_2 ," S.L. Hwang,* C.M. Hwang and I-W. Chen, at the 91st Annual Meeting of The American Ceramic Society, Indianapolis, IN, April 24-27, 1989.
26. "Effect of a Liquid Phase on Superplasticity of 2Y-TZP," C.M. Hwang,* S.L. Hwang and I-W. Chen, at the 91st Annual Meeting of The American Ceramic Society, Indianapolis, IN, April 24-27, 1989.
27. "Superplasticity in Mullite- ZrO_2 Composites," C.K. Yoon* and I-W. Chen, at the 91st Annual Meeting of The American Ceramic Society, Indianapolis, IN, April 24-27, 1989.
28. "Superplastic Forming of Fine Grained Ceramics," X. Wu* and I-W. Chen, at the 91st Annual Meeting of The American Ceramic Society, Indianapolis, IN, April 24-27, 1989.
29. "The Influence of Shear Coupling on Evaluating t to m Transformation in ZrO_2 Ceramics," K.J. Bowman* and I-W. Chen, at the 91st Annual Meeting of The American Ceramic Society, Indianapolis, IN, April 24-27, 1989.
30. "Grain Growth Control in Zirconia Polycrystals," S.L. Huang* and I-W. Chen, at the Fourth International Conference on the Science and Technology of Zirconia, Anaheim, CA, October 31-November 3, 1989.
31. "Stress State Effect on Transformation Toughened Zirconia," J.S. Cherng* and I-W. Chen, at the Fourth International Conference on the Science and Technology of Zirconia, Anaheim, CA, October 31-November 3, 1989.

32. "Transformation Textures in Zirconia," K.J. Bowman* and I-W. Chen, at the Fourth International Conference on the Science and Technology of Zirconia, Anaheim, CA, October 31-November 3, 1989.
33. "Fatigue of 3Y-TZP," S-Y. Liu* and I-W. Chen, at the Fourth International Conference on the Science and Technology of Zirconia, Anaheim, CA, October 31-November 3, 1989.
34. "An Elastic-Plastic Theory of Toughening of Brittle Solids," I-W. Chen, at the 92nd Annual Meeting of The American Ceramic Society, Dallas, TX, April 22-26, 1990.
35. "Superplastic Forming of Fine Grained Zirconia Ceramics and Composites," X. Wu* and I-W. Chen, at the 92nd Annual Meeting of The American Ceramic Society, Dallas, TX, April 22-26, 1990.
36. "Superplasticity of Fine Grained Alumina," L-A. Xue* and I-W. Chen, at the 92nd Annual Meeting of The American Ceramic Society, Dallas, TX, April 22-26, 1990.
37. "Creep-Enhanced Dynamic Grain Growth of Superplastic Zirconia," S.L. Hwang* and I-W. Chen, at the 92nd Annual Meeting of The American Ceramic Society, Dallas, TX, April 22-26, 1990.
38. "Grain Growth Control in Nonstoichiometric Oxides - Space Charge and Boundary Mobility," I-W. Chen* and S.L. Hwang, at the 92nd Annual Meeting of The American Ceramic Society, Dallas, TX, April 22-26, 1990.
39. "Grain Growth in Fluorite-Structured Oxide Solid Solutions," S.L. Hwang,* S. Rutter and I-W. Chen, at the 92nd Annual Meeting of The American Ceramic Society, Dallas, TX, April 22-26, 1990.
40. "X-ray Absorption Studies of Trivalent Oxide-Doped Ceria," P. Li,* I-W. Chen, T.Y. Tien, P.G. Allen and J.E. Penner-Hahn, at the 92nd Annual Meeting of The American Ceramic Society, Dallas, TX, April 22-26, 1990.
41. "Sr-Nb-O System: Electrical and Magnetic Characterization of Metallic Conductivity and Semi-conductivity," S.J. Keating,* I-W. Chen and T.Y. Tien, at the 92nd Annual Meeting of The American Ceramic Society, Dallas, TX, April 22-26, 1990.
42. "Sr-Nb-O System: Nonstoichiometry and Phase Equilibrium," S.J. Keating,* I-W. Chen, T.Y. Tien and Z.K. Huang, at the 92nd Annual Meeting of The American Ceramic Society, Dallas, TX, April 22-26, 1990.
43. "Fatigue Crack Growth and Life Time Prediction of Ceramics," S-Y. Liu* and I-W. Chen, at the 92nd Annual Meeting of The American Ceramic Society, Dallas, TX, April 22-26, 1990.

IX. CURRENT/PENDING SUPPORT

1. Fundamental Alloy Design of Oxide Ceramics and Their Composites (DOE-BES)
5/1/90 - 4/30/93, P.I.—I-Wei Chen
\$298,557
2. High Temperature Fatigue of Structural Ceramics (AFOSR)
10/1/90 - 9/30/93, P.I.—I-Wei Chen
\$615,845
3. Materials Processing and Engineering of Superplastic Ceramics (NSF)
8/1/91-7/31/94, P.I.—I-Wei Chen
\$270,000
4. Fundamental Experimental and Theoretical Studies on the Transformability and
Toughenability of Zirconia Based Ceramics (NSF)
5/1/92-4/30/95, P.I.—I-Wei Chen and J. Penner-Hahn
\$465,000

X. BUDGET

See attached DOE forms for budgets and budget explanation.

Budget attached.
As

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