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On

CERAMICS DERIVED FROM ORGANO-METALLIC POLYMERS

(GRANT NO. AFOSR-87-0107)

Submitted to

AIR FORCE OFFICE OF SCIENTIFIC RESEARCH

By

UNIVERSITY OF ARIZONA

Professor D.R. Uhlmann
Principal Investigator
July, 1988

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I. INTRODUCTION

Work carried out under the present ~~grant~~ was directed to several areas. All involved the use of organo-metallic precursors to produce novel ceramic and glass materials. The areas of principal activity included the following: (1) Polytetrafluoroethylene-silicate composites via sol-gel processing; (2) Fluoropolymer-modified silicate glasses; (3) sintering behavior of sol-gel derived anorthite and cordierite glass powders; (4) coating pretreatment effects in thermally nitrided sol-gel silica coatings; and (5) sol-gel derived coatings. Accomplishments in each of these areas will be summarized in the following sections.

II. PTFE - SILICATE COMPOSITES VIA SOL-GEL PROCESSING

Polytetrafluoroethylene (PTFE) is an organic polymer with a combination of useful properties. These properties include high chemical resistance, low surface friction, good thermal stability, and low dielectric constant. Unfortunately, PTFE is very difficult to process due to its high melt viscosity, low melt strength, and virtual insolubility; and it is soft and subject to cold flow.

Several processing and reinforcing technologies have been developed to overcome PTFE's deficiencies; and for decades it has seen wide use in a spectrum of applications. The developed technologies, however, have not solved all the problems associated with using PTFE; and many potential applications await further developments.

In the present study, an investigation was undertaken to determine the feasibility of producing PTFE-SiO_2 composite materials via an extension of the sol-gel process. The use of a sol-gel derived matrix offers the possibility of improving the processability and properties of PTFE-based components and coatings.

Bulk samples and coatings of PTFE-SiO_2 composite materials were made by combining PTFE dispersions with tetraethylorthosilicate (TEOS). The mechanism of PTFE incorporation into the glassy matrix was examined, as was the effect of different TEOS pre-hydrolysis conditions on composite microstructure. The lubricity of the composite coatings was also examined.

It was found that insoluble polymers such as PTFE can be incorporated into an alkoxide matrix to produce polymer/oxide composites. Vast differences in the roughness and morphology of bulk cast pieces result from differences in the processing conditions of the TEOS solutions. The causes of these changes involve interactions between the TEOS, solvent and surfactants used to disperse the PTFE particles.

STEM analysis has shown that PTFE particles are wet by the TEOS matrix, suggesting potential applications as adherent, low coefficient of friction coatings. The coefficient of sliding friction was reduced by incorporating PTFE into a TEOS coating; and heating was shown to help stabilize the lubricity. Further work is needed to optimize the coating composition and processing conditions to produce low friction, durable coatings.

III. FLUOROPOLYMER-MODIFIED SILICATE GLASSES

The sol-gel method provides a means for modifying glasses and crystalline ceramics with organic polymers. These new materials promise to have unique and varied combinations of mechanical, electrical and optical properties.

In work carried out under a previous AFOSR grant, polydimethysiloxane (PDMS) - modified SiO_2-TiO_2 glasses were prepared. It was found that, in these materials, the PDMS was chemically incorporated into the glass network. The chemical incorporation was assumed to occur by the condensation of terminal OH groups on the PDMS was prepolymer with the OH groups of hydrolyzed alkoxides. The results of thermal analysis were consistant with this mode of incorporation.

In the present work, an organic polymer which was not expected to react with glass precursor alkoxides was selected as the inroganic network modifier. Experiments were performed to determine the types of materials which could be made, and if the data from thermal analysis of these materials were consistent with previous theories of polymer incorporation.

A quantity of PVDF-PTFE copolymer was dissolved in methyl ethyl keytone (MEK). Specific amounts of copolymer solution were added to vials containing partially hydrolyzed TEOS. The quantity of copolymer solution was varied so that samples containing 10, 20, 30, 40, 50, 60, 70, 80 and 90 percent copolymer on a solute weight basis were obtained. The vials were sealed; and the samples were rolled for five hours. Additional samples were prepared which contained four types of prehydrolyzed TEOS and MEK with no copolymer.

Quantities of each sample and of each original TEOS solution were poured into open Al dishes and allowed to dry for twelve hours. Standard microscopic slides were also dip coated with ech solution in air, using a low torque, constant speed dip coater. Differential scanning calorimetry was performed at a heating rate of 10 C min^{-1} under N_2. Glass transition temperatures (T_g) were measured at the midpoint of the discontinuity in specific heat. Thermogravimetric analysis was performed at a heating rate of 20 C min^{-1} under N_2. Scanning electron micrographs (SEM) were taken of all coatings and monoliths.

The monliths made with acid catalyzed TEOS-coolymer solutions displayed a wide variety of appearances. In general, samples with 10-20% polymer formed opaque, white, brittle disks. There was obvious phase separation; and the bottom of the sample looked much different from the top. The samples with 30-50% copolymer also displayed some obvious phase separation; but it was much less than in samples with less copolymer. These samples were translucent, and did not crack upon drying. The samples with 60-70% copolymer showed even less obvious phase separation; but some was still apparent. These samples were very hard and tough, and could not be easily bent or broken. The samples with 80-90% copolymer were again transparent; and no obvious phases separation was apparent. The samples could be bent.

The monoliths made with base catalyzed TEOS-copolymer solutions displayed a wide but different variety of appearances than those made from acid catalyzed TEOS-copolymer solutions. In general, samples with 10% polymer formed cracked, opaque, white chunks. The samples with 20-40% copolymer formed uncracked, opaque, white disks which were brittle. The samples with 50-90% copolymer formed translucent disks which curled upon drying, much like the pure copolymer. Obvious phase separation was observed in some, but not all of the samples. The brittleness of these samples decreased as the proportion of copolymer increased.

The melting point of the pure copolymer was measured as 122 C, which is within the 122-126 C published range. All the copolymer-modified glass samples, with the exception of one, displayed some distinct melting points within this range. This indicates that in all samples, there were regions of unrestricted copolymer.

The pure copolymer also displayed a distinct glass transition around -98 C. A transition near this temperature was also observed in most of the copolymer-modified glass samples. A second glass transition, which was not observed in the pure copolymer, was seen in about half of the copolymer-modified samples. This transition occurred between 40 and 66 C in samples with < 50 wt.% copolymer.

The weight loss transition of the pure copolymer began at 457 C and was substantially complete at 525 C. At 600 C, the residue was only 3.2 wt.%. Addition of the four TEOS solutions to the copolymer substantially changed its degradation behavior. For example, the weight loss transition for the sample derived from the solution containing 30% acid catalyzed TEOS and no ethanol did not begin until 498 C.

These results, coupled with the structural observations carried out using scanning electron microscopy, indicate that a new class of materials has been created. These materials have intriguing combinations of properties, as well as interesting structural characteristics. They are representative of the broader class of organic-modified oxides, which are receiving continued attention in our laboratory.

IV. SINTERING BEHAVIOR OF SOL-GEL DERIVED ANORTHITE AND CORDIERITE GLASS POWDERS

A significant portion of the research into sol-gel or wet chemical techniques for synthesizing ceramic materials is motivated by the electronics industry in an effort to manufacture devices with superior properties or with more cost-effective processing. Research by our group into sol-gel derived materials for use as ceramic substrates has focussed on the alkaline earth alumino-silicates Ccordierite ($2MgO\cdot 2Al_2O_3\cdot 5SiO_2$) and Anorthite ($CaO\cdot Al_2O_3\cdot 2SiO_2$) because of their relatively low values of dielectric constants (~5) and thermal expansivity. Work carried out previously with AFOSR support demonstrated the feasibility of generating amorphous powders of the Anorthite and Cordierite compositions using an all-alkoxide approach. The sequence of phase development upon crystallization has also been documented. The objective of the present work was to explore the densification behavior of these materials and to investigate the applicability of the Frenkel-Scherer sintering model to the observed phenomena.

The synthesis technique used to generate the powders involved partially hydrolyzing tetraethyl orthosilicate, freezing it, and then adding it to cold solutions of the double alkoxides $Ca(Al(OEt)_4)_2$ or $Mg(Al(OEt)_4)_2$. After heating to room temperature the precursor solution was slowly poured into ammoniated water to induce full hydrolysis and effect precipitation. The precipitate was filtered, rinsed successively in EtOH and H_2O_2, dried at 100 C and then calcined by heating at 1 C/min to 700 C in dry O_2.

The resulting powder was x-ray amorphous. STEM and SEM analysis indicated that the powder consisted of agglomerates with sizes ranging to as large as 100 microns. For Anorthite, the majority of primary particles had diameters about 100 Å with some small fraction having diameters about 400 Å.

The Cordierite primary particle diameter was 50-100 Å.

It was found that Anorthite and Cordierite glasses with densities greater than 96% can be produced by sintering sol-gel derived powders at temperatures less than 900 C. Small pore sizes (less than 100 Å) allow the samples to densify in reasonable times even for "apparent" viscosities in excess of 10^{14} poise. During densification, pellet viscosities increase by over an order of magnitude. The time dependence of this increase has a characteristic time on the order of that required to densify the samples. The viscosities derived from the sintering data for Anorthite and Cordierite have higher activation energies than those of melt-derived liquids of the same composition. The magnitudes of the viscosities derived from sintering data exceed those of melt liquids by factors of 4 (for Anorthite) to 110 (for Cordierite).

V. COATING PRETREATMENT EFFECTS IN THERMALLY NITRIDED SOL-GEL SILICA COATINGS

The high dielectric strength of oxynitride thin films has created interest in these materials for use in a number of electronics applications. Conventionally, oxynitride films have been made on silicon by nitriding thermally grown oxide films at high temperature (900 - 1000 C) in ammonia. These films, however, generally exhibit depleted nitrogen contents in the bulk of the film as a result of the difficulty of diffusing the reacting species through the nitrided surface. To overcome this problem, sol-gel processing techniques, wherein a porous silica film is applied to the silicon substrate and then nitrided, have been attempted. The average decrease in diffusion length provided by the porous films has been successful in producing homogeneous thin films (~50 nm when fired) on silicon substrates.

In addition to potential in the electronics field, oxynitride glasses have shown promise for use in mechanical applications as a result of their extremely high hardness and strength. As coatings, oxynitride glasses could provide greatly improved abrasion resistance, as well as strengthening to glass bodies. It is envisioned, however, that to provide significant abrasion resistance, such coatings would need to be much thicker than 50 nm. As a result, the objectives of this work were to produce thicker oxynitride coatings, to alter processing parameters to optimize the homogeneity of nitrogen incorporation, and to investigate the effects of using glass (as opposed to Si) substrates.

Coating solutions were prepared by partially hydrolyzing tetraethoxysilane (TEOS), 2 moles H_2O per mole TEOS, diluted 1:1 in ethanol. The reaction was catalyzed by adding HCl to bring the solution pH to 3. The solution was stirred under argon, in a water bath maintained at 20 C.

Both fused silica glass and silicon were used as substrates. Both dip and spin coating were used. The coated samples were dried for 10 min. under a heat lamp, and diced into 1 cm squares. Both coating techniques produced 240-280 nm coatings which were dried at various temperatures and then fired at 1050 C in flowing ammonia for one hour.

Coating thicknesses were determined by profilometry. Surface composition was determined by x-ray photoelectron spectroscopy using Mg anode. Auger sputter depth profiling was used to determine the homogeneity of the nitrogen distribution.

It was found that relatively thick (>1200 Å when fired) homogeneous silicon oxynitride coatings can be produced by the high temperature nitridation of porous silica films in ammonia. While the surface nitrogen content of these films was mildly enhanced by drying the coatings at high temperatures before nitriding, the homogeneity of the nitrogen content was drastically decreased by prefiring heat treatments. The substrate composition (silica or silicon) was shown to have little effect on the nitrogen incorporation. However, the coated silicon substrates showed a much greater tendency towards cracking, especially in the case of the lower drying temperatures needed to achieve homogeneous nitrogen incorporation.

For use as an abrasion resistant coating for glasses and ceramics, coatings thicker than 120 nm (fired) will be necessary. It does not appear, so far, that there is any intrinsic limit to the thickness of coatings which can be homogeneously nitrided, although the sensitivity of the homogeneity to drying schedules observed in the present work indicate that minor processing changes become important as coating thicknesses increase. In the present work, the thickness of homogeneous oxynitride films was limited only by the ability to produce thick, crack-free dry precursor coatings.

VI. SOL-GEL DERIVED COATINGS

The utility of sol-gel for producing glass and ceramic materials with high purity, novel compositions, novel microstructures and potentially high chemical homogenity at relatively low temperatures (compared with conventional processing) is by now well recognized - see, e.g., (the many papers on this topic at the present Conference. Considerable attention has been (and is being) directed to the use of sol-gel techniques in the production of monolithic bodies. The relatively high cost of precursors coupled with problems associated with drying stresses and the effective removal of volatiles, and in come cases the use of relatively expensive processing methods, seem likely, however, to limit the widespread use of such techniques in the direct production of bulk glasses and ceramics.

The most natural applications of sol-gel methods appear to lie in the production of coatings and powders.

Sol-gel derived coatings offer outstanding opportunities because the attractive features offered by such processing can be obtained without many of the disadvantages cited above. In the case of coatings, the material cost (and hence precursor cost) is relatively unimportant; rapid drying can be empolyed without the development of cracking; shrinkage is relatively uniform (normal to the substrate); and the elimination of volatiles is comparatively easy. Further, rather simple and commercially viable processing methods- such as dip and fire, spin coating, spraying and roller coating - can be used to coat substrates of nearly any size and geometry.

Sol-gel coatings have already been used to good advantage in a wide variety of applications. These include transparent coductive coatings, passivation coatings, optical control coatings, antireflection coatings, porous coatings, adhesion-promoting coations, and coatings used in a number of mechanical and electrical (electronic) applications.

A. SELECTED APPLICATIONS OF SOL-GEL COATINGS

(a). Transparent Conductive Coatings: Such coatings are used in a wide variety of applications, from displays to thermal de-icing of windows. Most attention has been directed to materials based on the oxides of In and Sn, which are n-type degenerate semiconductors with attractively low resistierties (10^{-4}-10^{-2} Ωcm) and relatively wide band gaps (2.3 - 4.6 eV). Carriers are introduced via oxygen deficiency or chemical doping. Examples of the latter include F of Sb with SnO_2 and Sn with In_2O_3. The last represents the widely used indium tin oxide (ITO) system.

Sol-gel syntheses of transparent conductive coatings have included cadmium stannate (ITO), and Sb-doped SnO_2. Attractively high transparencies and electrical conductivities have been obtained, particularly with the latter two materials. Post-deposition heat treatments, typically in the range of 400-500°C in controlled atmospheres, are used to develop desired crystalline microstructures, stoichiometries and properties, the coatings are reported to have good chemical and adhesive characteristics.

(b) Optical Control Coatings: The most notable examples of such coatings are the IROX coatings which include colloidal Pd particles in dip-coated TiO_2. films. Effective use is made of the ambient atmosphere to effect hydrolysis of the Ti alkoxide solutions on the substrate glass (the rate of condensation of TiOH groups is very rapid). Besides the desired optical characteristics, these coatings have good scratch resistance and environmental stability.

(c) Antireflection Coatings: The use of coatings to reduce reflection losses at surfaces has been extensively used for more than half a century. Applications range from optical components and CRT face plates to solar cells and glass picture frames. Both single layer and multiple (double) layer coatings have been developed using sol-gel techniques, and offer promise for technological use.

In typical single layer (SLAR) coatings, the index of refraction is varied smoothly from the free surface to the substrate and the thickness of the coating is controlled. These coatings have interconnected pores whose dimensions are controlled by the composition, size and morphology of the polymers in solution prior to deposition. The porosity (and hence the index) can be further modified by leaching and thermal treatments. Alkoxide-derived SLAR coatings have reduced the reflectivity of glass surfaces to less than 1.4%; and simple coatings based on Aerosil particles and Si alkoxides have reduced reflectron by 60%.

While SLAR coatings can reduce the reflectivity to ~zero at a particular wavelength, they are ineffective in maintaining low reflectivities across a broad range of wavelength. Such problems are overcome by use of multilayer coatings, which are typically deposited from the vapor in commercial practice. Double layer AR coatings based on sol-gel derived SiO_2 and TiO_2 and having attractive anti-reflection characteristics over a range of wavelengths, have been prepared.

(d) Passivation Coatings: Coatings have been extensively used to protect bodies from their environments and to protect the environments from constituents in the bodies. The potential for the commercial application of such coatings obtained by sol-gel methods seems outstanding, taking advantage of the low processing temperature offered by sol-gel techniques and the potential for good adhesion to substrates. In many applications, issues of pinholes and porosity will be important. An example of coatings used to protect the external phase from constituents in the substrate is provided by the use of SiO_2 layers between ITO coatings and sodalime-silicate glass substrates in conductive coated glass for display applications.

(e) Porous Coatings: Sol-gel processing offers the opportunity of developing coatings with tailored pore size distributions, total volume of pores and tailored pore wall chemistries. Such coatings have been explored with reference to catalytic applications and membranes for ultra filtration in addition to the antireflection coatings discussed above.

The utility of sol-gel coatings in such applications will depend on the ability to tailor effectively the chemistry and size distributions of the pores and to provide desired mechanical integrity. Other uses of porous coatings include their application as reaction media. Recall as an example the reaction of highly porous silica coatings with NH_3 to produce oxynitride coatings. The potential for producing novel coatings via vapor phase or liquid phase infiltration of porous sol-gel derived coatings and subsequent reaction seems considerable.

(f) Adhesion Promoting Coatings: Sol-gel coatings offer attractive opportunities in the area of adhesion formation. Rather than attempt to bond a polymer, e.g., to a difficult-to-adhere metal oxide surface, it seems promising to coat the surface with a different composition which is adherent to the substrate but to which the polymer can readily bond. Coating-substrate adhesion is of critical importance in such applications, which appear at present to be substantially unexplored.

(g) *Coatings in Mechanical Application:* The mechanical properties of coatings, including their hardness and abrasion resistance, are important in many applications. In addition sol-gel derived coatings can be used to provide materials with desired bulk properties and improved resistance to abrasion and/or increased strength. Improved abrasion resistance can be provided by hard coatings such as oxynitrides; and increased strength by the coatings filling in flaws on the surfaces of the bodies and in some cases by their providing compressive stresses at the surfaces via $\Delta\alpha$ between substrate and coating (analogous to the familiar glazing techniques).

A striking examples of strengthening glass by sol-gel coatings was provided by work in our laboratory. Studying SiO_2 coatings on SiO_2 rods, increases in strength exceeding 2X compared with uncoated rods given the same thermal treatment were observed. Subsequent work yielded increases in strength in the range of 3X. Since the coatings are expected to be in a state of tension, the strengthening does not involve surface compression. Rather, it almost certainly involves filling-in the surface flaws and forming chemical bands between the coating and the walls of the flaws. Use of borosilicate coatings on SiO_2 substrates have yielded coatings which can withstand tensile stresses in the range of 30 ksi. Application of low expansion coatings such as alkali borosilicates on higher expansion substrates should provide flaw-filling and surface compression.

To provide long-term increases in strength under service conditions, it seems important to develop coatings which are abrasion-resistant as well as flaw-filling (and which ideally have lower expansion coefficients than the substrates as well). In this regard, oxynitride and perhaps oxycarbide coatings appear to offer outstanding promise. The work described in Section V above addresses many of the issues involved in the effective use of such coatings.

The use of sol-gel derived coatings to improve the mechanical performance of materials seems to offer considerable promise. Important in many cases will be the ability to provide thick coatings with an efficient process. It should also be noted that the flow-filling process is not effective with very small flaws, and hence that the strengthening by this process alone is limited to the range of 30 ksi or so. Application of hard coatings to strong, freshly formed glass and ceramic bodies seems attractive for preserving the initial high strengths.

(h) Coatings in Electrical (Electronic) Applications: The potential electrical and electronic applications of sol-gel technology are wide-ranging; and use of this technology in the production of powders represents established commercial practice for materials such as $BaTiO_3$ and ferrites. The use of sol-gel derived coatings in such applications is relatively still in its infancy.

Among the films and coatings of interest in this area which have been prepared by sol-gel methods, the following are cited as examples:

(1) Ta_2O_5 and doped Ta_2O_5: These materials, prepared by anodic oxidation of the metal, are widely used in precision capacitors, and have potential for use as storage capacitor dielectrics in high density dynamic RAM devices. Coatings prepared from Ta alkoxide solutions have been developed under a previous grant from AFOSR; and their transformation behavior and dielectric characteristics have been determined. For the pure oxide, dielectric properties similar to those of anodically grown films are obtained

after firing to effect densification and crystallization (in detail, the dielectric constants are somewhat smaller for the sol-gel derived films). The great potential here of sol-gel techniques seems, however, to lie in the preparation of doped films with tailored dielectric properties.

(2) $PbTiO_3$, $PbZrO_3$, PZT and PLZT: These materials are characterized by high dielectric constants and have applications ranging from capacitors and piezoelectric devices to optical modulators and electrooptic shutters. Sol-gel syntheses of films of these materials have been carried out by several research groups, generally employing mixtures of salts and alkoxides in solution, in some cases reacting them to form complex alkoxides.

(3) Spin-on glasses: These materials, most often thin films of SiO_2 deposited from an alcohol solution and densified with high temperature treatment, have wide use in integrated circuit fabrication. The coating solutions are typically hydrolyzed alkoxides of Si, and in some cases of Ti. Such films have generally been used with Si wafers, and lately have found use with GaAs. The latter case seems to offer notable opportunity, since it lacks a native oxide; but full utilization of the approach will likely depend on the development of chemically more complex coatings with tailored properties.

III. CRITICAL ISSUES IN SOL-GEL COATINGS

The use of sol-gel techniques in the preparation of coatings has been seen to have a wide range of potential, and in some cases already achieved, applications. The achievement of anything like the full potential of these techniques will depend upon progress in a sizable number of areas. These include: (a) coatings of uniform thickness; (b) multicomponent coating compositions; (c) thick coatings; (d) composite coatings; (e) shelf life, pinholes and structural homogeneity; and (f) non-conventional oxides and non-oxides.

In summary, the field of coatings represents a veritable land of opportunity for sol-gel technology. Actual and potential applications are bountiful; and the landscape is replete with unexplored areas from which exciting developments are almost certain to emerge. Indeed, there are so many potentially important scientific questions and technological applications that one has difficulty in selecting the best topic for study. The field of coatings also avoids many of the problems, both technical and economic, which plague the use of sol-gel methods in the production of monoliths.

Achievement of even a small fraction of the potential of sol-gel derived coatings will have a tremendous impact on technology across a broad front. Such achievement will require not only greatly expanded interaction between chemists and materials scientists, but also much closer collaboration between these scientists and those with expertise in areas such as optics and electronics. We live in a multidisciplinary age; and few areas offer greater payoff for interaction across disciplines than that of sol-gel derived coatings.