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PROPERTIES OF MOLTEN CERAMICS

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INTRODUCTION

Temperatures which approach or exceed the melting point impose severe conditions on ceramics. Kinetic effects involved with compatibility and phase equilibria are enhanced. Experimental problems are compounded. This primarily is the reason why few property measurements have been made of the behavior of liquid refractory ceramics. However, there is a rapidly increasing need for this information, both from a practical standpoint and from a basic understanding of the liquid state of molten ceramics.

An understanding and control of the properties of the liquid ceramic should result in improved ceramic processes and better ceramic products and in more realistic predictions of behavior where the temperature of ceramic articles exceeds the melting point. For example, the development of fusion casting and reinforced ceramic composites would benefit if the surface tension, viscosity, and volume change on solidification were known; processes for growing single crystals and filaments of ceramics from the melt could be improved.

Fundamentally, precise measurements of liquid properties are the basis for understanding the structure of the high temperature liquids. Properties such as expansion, viscosity, compressibility, and thermal diffusivity must depend upon structural parameters. Measurement of thermal expansion, density, surface tension, and compressibility relate to the thermodynamic properties of liquid ceramic materials.

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Except for glass formers, such as SiO_2 and B_2O_3 , reliable data for crystalline ceramics in the liquid state is almost nonexistent. In contrast, a large amount of data is available for non-oxide ionic salts which melt at relatively low temperatures. (1,2)

Techniques have been developed for measuring the volume change on melting, density, melting point, expansivity, surface tension, viscosity, adiabatic compressibility, and thermal diffusivity of molten ceramic materials to 3000°C . Many of these techniques are extensions of general methods (3) used to measure liquids at lower temperatures, with modifications to reflect the severe conditions imposed by the high temperatures. Some of the techniques successfully used by the authors to study molten ceramics are described. The associated experimental difficulties and representative results are also presented. Because of space limitations, no attempt has been made to present comprehensive review of the potential high temperature, experimental methods or an analysis of the results.

RADIOGRAPHIC TECHNIQUE

X-radiography of the molten ceramic in an electron beam sealed refractory metal capsule is used to observe the volume and surface properties of the liquid. The expansivity, density, and volume change on melting are determined by observing the volume of the materials. The surface tension is determined from the shape of the meniscus or the pendant drop which is formed.

The radiographic system is illustrated schematically in Figure 1. The sample is radiographed with a 300-kv X-ray source. An X-ray image intensifier and vidicon camera are used to monitor the sample continuously, and film is inserted for a permanent record. A tungsten-mesh resistance heating element surrounded by split radiation shields allows X-radiography under effectively isothermal conditions with a minimum of absorbing material in the image-forming X-ray beam. The furnace can be operated with an inert or reducing gas or in vacuum.

The large density change on melting for a material such as Al_2O_3 allows direct radiographic observation of the volume change on melting, meniscus, and expansion of the liquid, Figure 2. This figure also illustrates the ability to observe the liquid-solid interface in a partially melted sample made on a capsule which was heated with the midpoint approximately 100°C hotter than the ends. Melting started inward from the wall at the midpoint. The liquid-solid interface is clearly visible, as are two bubbles which formed in the liquid. Precision film densitometry could possibly be used to determine the relative density from these nonisothermal radiographs.

Fig. 1. Facility for radiographic measurement of liquid density,

Fig. 2. Radiograph of Al_2O_3 showing partial melting and volume change and density of liquid UO_2 .

SURFACE TENSION

Surface tension is being measured by two liquid surface shape techniques: (1) pendant drop shape, and (2) equilibrium meniscus shape. Pendant drops are formed on the end of solid oxide rods which are suspended in the capsule such that the free end of the rod coincides with the maximum of the temperature gradient along the capsule. An appropriate temperature gradient is established by resistively heating a capsule machined with a variable wall thickness to provide a resistance gradient. In this manner, only the end of the rod is melted and a stable pendant drop is formed. Several drops can be formed from each rod by gradually increasing the temperature. As more material becomes molten, the drop increases in size, becomes unstable, falls, and a new drop is formed. The shape of the meniscus which forms in the refractory metal capsule when the oxide becomes molten can also be used to obtain the surface tension.

A major problem in obtaining accurate surface tension values is the effect of impurities. In small concentrations, impurities which are not surface active have a negligible effect on the surface tension. However, a surface active species can markedly change the surface tension value. The pendant drop technique is useful in that the molten drop is in contact only with a solid of the same nominal composition as the drop. This removes the possibility of the surface tension being affected by the presence of impurities picked up from a supporting material. Measurement is limited, however, to the value at the melting point. In contrast, the capillary meniscus method yields surface tension values as a function of temperature but these values are potentially affected by contamination from the crucible material.

The surface tension is determined from an analysis of the shape of the drop or the meniscus using a computer program.⁽⁴⁾ The program requires a measurement of the coordinates of the drop or meniscus profile. With the drop coordinates and the density of the liquid, the surface tension can be calculated utilizing the technique developed by Bashforth and Adams.⁽⁴⁾

Results of the surface tension and density of molten Al_2O_3 , MgAl_2O_4 , Sm_2O_3 , and UO_2 are shown in Table I. Measurements of the surface tension and density of molten Al_2O_3 have been reported utilizing other techniques.⁽⁶⁻¹⁰⁾ Our results agree very well with these.

DENSITY

The density of the molten ceramic material is calculated from the known weight of the sample inserted into the capsule and its

TABLE I. Surface Tension and Density of Molten Ceramics

Material	σ dynes/cm	ρ g/cm ³
Al ₂ O ₃	574 $\pm$ 68 pendant drop	5.632-1.127 $\times 10^{-3}$ T for T > 2333°K
	360 $\pm$ 40 meniscus, Mo capsule	
	574 $\pm$ 68 meniscus, W capsule	
MgAl ₂ O ₄	481 $\pm$ 102 meniscus	5.324-9.865 $\times 10^{-4}$ T for T > 2403°K
	490 $\pm$ 98 pendant drop	
Sm ₂ O ₃	962 $\pm$ 53 pendant drop	5.9
UO ₂	441 $\pm$ 35 meniscus	8.7

volume as a function of temperature. The volume is calculated by measuring the height of the liquid column from the radiographs and using the inside diameter of the refractory metal crucible after correcting for thermal expansion. Corrections also are made for the volume of the material contained in the meniscus.

VOLUME CHANGE ON MELTING

The volume change on melting is determined by extrapolating the measured volume of the solid as a function of temperature to the melting point and extrapolating the volume of the liquid as a function of temperature to the melting point. The difference between these two volumes is the volume change on melting. Results of measurements on the thermal expansion and volume change on melting of UO₂ are illustrated in Figure 2.

Several radiographic measurements of change in volume on melting and liquid expansivity of single crystal sapphire were made using molybdenum and tungsten capsules. In each experiment, a hysteresis in liquid volume was observed as the specimen was heated and cooled above the melting point. In one case in which the tungsten capsule was heated in flowing argon to 2800°C, the volume change on solidification was approximately 24% compared to 33% on melting and the expansivity on cooling was correspondingly greater than the expansivity on heating. This was found to be due to the presence of bubbles during the initial melting. The decrease in specific volume with time and temperature above the melting point indicates that the gas can be removed from the liquid. A sufficiently long refining period results in apparent specific volume values approaching the true value.

VISCOSITY DATA		
	TEMPERATURE °C	VISCOSITY, POISE
UO ₂	2755	0.46
	2795	0.36
Al ₂ O ₃	2070	1.38
	2170	0.746
	2260	0.648
	2370	0.584
	2475	0.565
	2575	0.505
	2675	0.480
MgAl ₂ O ₄	2185	0.117
	2290	0.115
	2385	0.112

Fig. 3. Viscosity apparatus and viscosity data for Al₂O₃, UO₂, and MgAl₂O₄.

Fig. 4. Ultrasonic velocity measuring apparatus for high temperature liquid ceramics.

Fig. 5. Adiabatic compressibility and ultrasonic velocity for molten Al_2O_3 .

VISCOSITY

The kinematic or shear viscosity of molten ceramics can be measured with an oscillating cup viscometer. (3,13-14) (Figure 3) This technique consists basically of measuring the time rate of decrease in the amplitude of a free-oscillating, closed capsule (containing the liquid) suspended from a torsion wire. The amplitude decrease (or damping) results from both the viscous motion of the liquid and the internal friction of the torsion wire. The wire is chosen such that its damping is small compared to the damping of the system.

The amplitude changes can be measured by a variety of electronic, mechanical, or optical methods. A convenient method is to view the oscillating scale with a television system and replay a video-tape record of the graduated scale in stop action mode. The total period of a number of successive oscillations is measured with a stop watch.

Viscosity values are calculated from the measured damping and period, the known values of the moments of inertia of the sample and pendulum, the liquid density and the crucible geometry.

For high temperatures, the sample is contained in a vacuum welded, refractory metal capsule and suspended from a W or W-Re torsion wire inside a W mesh resistance heated, vacuum furnace. The system is started oscillating to a determined amplitude and period using an electromagnet and pendulum which is placed above and outside the hot zone. Attached to the pendulum is a mirror and graduated scale to be used to measure the damping. The magnetic cocking system must be precisely balanced to prevent any lateral swing of the capsule. Both the capsule and the torsion system must be operated in a vacuum.

Temperatures are measured with an optical pyrometer sighted on the rotating capsule and with a W-5% Re/W-26% Re thermocouple positioned near the bottom of the rotating capsule. Both temperature measurements are difficult because of non black-body conditions and the distance between the capsule and the thermocouple. However, if the melting point of the sample is known, an internal check of temperatures is obtained.

The volume of the liquid ceramic must not completely fill the capsule nor should any of the meniscus reach the top of the capsule. Consideration is also given to the viscous drag on the bottom.

It is essential that the capsule dimensions and sample height be chosen so as to optimize the results. A very low viscosity requires a larger sample diameter in an effort to reduce the damping rate. Thus, a great deal of trial and error is involved

in choosing the proper crucible size, the sample height and type, and size of torsion wire.

The viscosity has been measured for a number of oxides including Al_2O_3 , UO_2 , and magnesium aluminate spinel. The results are shown in Figure 3. For Al_2O_3 , the viscosity decreases linearly from 0.75 poise above the melting point to 0.5 poise at 2700°C . The viscosity of UO_2 was measured just above the melting point. Assuming a liquid UO_2 density of 8.7 g/cm^3 , the calculated viscosity values at 2755°C and 2795°C were 0.46 and 0.36 poise. Note that the observed melting temperature is $50\text{--}100^\circ\text{C}$ less than other reported values which illustrates the difficulty in measuring absolute temperatures. Other investigators report values from 0.5 to 10 poise. (14-16)

The viscosity of spinel (MgAl_2O_4) was significantly less than the viscosity of Al_2O_3 and exhibited a very small temperature linear dependence from 2185 to 2385°C , 0.117 to 0.112 poise, respectively.

ADIABATIC COMPRESSIBILITY

The adiabatic compressibility of molten ceramics is determined at high temperatures by measuring the ultrasonic velocity of longitudinal waves in the liquid contained in a closed capsule. This interference technique, used quite extensively for low melting ionic salts, (2) silicates, (17) and molten rock, (18) uses a transmission rod to transmit an ultrasonic pulse from the cold transducer to the liquid. The method was modified to measure the liquids of molten oxides. The electrical signal is applied to a quartz transducer which acts both as the transmitter and receiver. The 18 MHz longitudinal waves propagate along a 40 cm long, 1.9 cm diameter tungsten single crystal to and from the molten oxide sample.

The molten oxide is contained in a polycrystalline tungsten crucible which is vacuum welded to the end of the crystal transmission rod, Figure 4. The crucible cavity is machined with parallel surfaces and contains an expansion plenum to allow for the volume change and expansion of the molten oxide. The sample, generally a single crystal, is machined to a size such that the oxide nearly fills the crucible just below the melting point. Upon melting, the specific volume increases, filling the cavity and covering the reflecting surfaces with molten sample. The excess liquid is allowed to fill the expansion chamber above and on the side of the crucible so as not to interfere with the propagation of sound waves. Scattering slots are machined into the top of the crucible to prevent unwanted echoes from being reflected back from the top of the crucible.

The crucible and sample end of the transmission rod are heated in the tungsten mesh, resistance heated vacuum furnace. The transmission rod extends from the hot zone of the furnace through O-ring seals and is externally water cooled.

Temperatures are measured using an optical pyrometer sighted in a black-body hole drilled in the side of the crucible. In addition, a W-5% Re/W-26% Re thermocouple is centered directly above the crucible. The system is calibrated using the observed melting point of the Al_2O_3 (2050°C). The thermocouple temperature is consistently lower than the melting and pyrometer temperature but is used conveniently to control the furnace temperature.

Upon melting, the sound waves propagate along the W crystal to the molten sample, pass through the liquid and reflect from both surfaces of the tungsten crucible in contact with the molten oxide. The phase comparison of the transmission and reflected waves is measured from which the velocity of sound in the liquid can be calculated.

The adiabatic compressibility (β) of the liquid is calculated from the relation

$$\beta = (\rho v^2)^{-1}$$

where ρ is the density and v is the longitudinal sound velocity.

The results for Al_2O_3 are plotted as a function of temperature in Figure 5.

THERMAL DIFFUSIVITY

The laser pulse, thermal diffusivity technique, generally used to measure the diffusivity of solids, was modified to measure heat conduction of molten materials. The pulse method(20-21) consists of heating one surface of a thin sample disc with a short heat pulse from a laser. The heat pulse passes through the sample and the temperature transient on the opposite surface of the sample is measured (Figure 6A). The temperature change is recorded and the thermal diffusivity is determined from the shape of the temperature-versus-time curve.

For the technique to be valid (1) the energy pulse must be uniformly absorbed over the front surface and at the surface only; (2) the laser pulse time must be short compared to the time for the heat pulse to pass through the sample; (3) heat flow must be normal to the heated surface; and (4) no heat must be lost from the sample during the measurements. If these conditions are met, the thermal diffusivity (α) can be determined from the sample thickness and time-temperature curve.

LIQUID SAMPLE HOLDERS

Fig. 6. Thermal diffusivity apparatus and sample holders for liquid ceramics showing results for Dresser basalt.

Fig. 9. DTA results for Pm_2O_3 - Sm_2O_3 binary system.

coupled with a potential high sample vapor pressure, can cause excessive swelling of the sealed metal container.

If the vacuum-sealed capsule is measured in gas atmosphere, permeation of the gas through the container can occur. Choice of cover gas can minimize this problem.

Temperature measurement and calibration become increasingly more difficult as the temperature increases. It is impractical or impossible to measure the temperature of the liquid directly. Use of thermocouples is complicated by loss of electrical resistance of insulators, diffusion in thermocouple, reactions with the system and lack of calibrated emf's. This problem is partially solved by using self-supporting, bare refractory metal thermocouples in the hot zone of the furnace. Optical pyrometer measures are generally made simultaneously with thermocouple measurements as a consistent check of temperatures. Temperature calibration is difficult yet the system can be self calibrating for samples with known melting points since melting is easily observed.

For high temperature measurements, the sample is heated in a tungsten mesh resistance furnace in vacuum, inert, or reducing atmospheres. The specimen, approximately 1.0 cm in diameter and 0.05 to 0.15 cm thick, is supported in a ceramic holder which is positioned in the hot zone of the cavity on a 1.4 cm tungsten tube. The vertical arrangement minimizes the loss of optical alignment which is essential to accurate measurements.

The temperature transient on the back surface of the sample is measured with an optical detector such as a liquid N₂ cooled, indium antimonide detector which is focused on the sample surface. The output from the detector is amplified, displayed on an oscilloscope and photographed on film. Temperatures are measured and the furnace controlled with a W-5% Re/W-26% Re thermocouple set in a hole in the sampleholder. The high temperatures require a self supported bare-wire thermocouple. An optical pyrometer is also used to measure the temperatures of the back surface.

Measurements are generally made using small solid discs. However, to measure liquids, a sample holder is required to contain the liquid in the disc shape, Figure 6C. Two methods were used, the choice of which depends upon the opacity of the sample, reactions with the sample crucible, the temperatures required, and the properties of the liquid at high temperatures.

Single crystal Al₂O₃ can be used to contain opaque materials. Since sapphire is transparent to the laser beam and infrared detector, even at high temperatures, the sample if opaque could be heated directly with the laser (Figure 6). The crucible is designed with polished surfaces on top and bottom and has expansion plenums to accommodate the increase in sample volume and for gas release on melting.

Thin melt crucibles of platinum or refractory metals can also be used (Figure 6C). However, a good thermal contact between the metal and the sample is necessary. The conductivity of the container must be large compared to the sample. Conversely, the crucible wall must be thin to prevent thermal shunting.

At the high temperatures above the sample melting point, the sample can wet the crucible and flow over the top edge of the crucible. This problem can be remedied by placing a small lip on the rim of the crucible. However, this does increase the chance of error due to thermal shunting.

This technique has been used to measure the thermal diffusivity of basalt. Measurements were made of Dresser and Columbia River basalt from room temperature to 1600°C. The results for Dresser basalt are illustrated in Figure 6B. The thermal diffusivity of the as-received rock decreases with increasing temperatures similar

to that of oxide insulators. The results for the Dresser basalt were not reversible with the diffusivity being significantly lower on cooling and during subsequent measurements. This irreversibility indicates the permanent change from a relatively crystalline basalt to a more vitreous body after melting. The results for both basalts show a sudden decrease near the melting temperature which was followed by a slow increase in the molten state.

The data for the fused basalt using the platinum crucible without the lip are in good agreement with the measurements made using the sapphire crucible. However, the addition of a large lip, necessary to prevent surface movement of the liquid, resulted in apparent higher values. At temperatures above 1150°C , transparency of the basalt to the laser and infrared detector again became a problem. This may have accounted for the sudden increase in diffusivity above the melting point. Thicker samples appeared to minimize this problem.

Although this technique appears to provide an easy and rapid method for measuring the thermal diffusivity of liquids, there are problems which must be resolved, most of which relate to the maintenance of the thermal boundary restrictions, with the reactions between the molten sample and the crucible and with the unusual properties of the liquid, i.e., surface tension.

MELTING POINTS AND PHASE EQUILIBRIA

A high temperature differential thermal analysis apparatus is used to measure melting points and phase equilibria to 3000°C . The equipment is patterned after that of Rupert(22) Although essentially the same as conventional DTA apparatus, unusual features make it particularly suited for use at these high temperatures (Figure 7). A most unusual aspect is the use of reverse-biased, silicon photo-diodes for temperature sensors. The output is amplified in the thermal analysis circuit and can be displayed in a number of ways. The temperature of the sample and/or standard, the temperature derivative, or temperature difference can be plotted against temperature, time, or derivative of temperature on a recorder or oscilloscope. Conventional DTA plots can be obtained and ΔT 's of a few tenths of a degree are measured at 2000°C .

A RF induction generator heats the graphite or refractory metal block (1.9 cm diameter and 1.9 cm high) using a current concentrator, (Figure 8). Cavities, 0.64 cm in diameter and 0.64 cm deep, contain samples weighing a few tenths of a gram. A lid, with 0.150 cm diameter holes for the detectors to see the samples, covers the block. The block and concentrator are inside

Fig. 7. Block diagram of high temperature DAT apparatus.

Fig. 8. Sample block, lid, and heat shield for high temperature DTA.

a fused quartz container in vacuum or inert or reducing gas. Greater sensitivity is obtained in vacuum. The choice of block material was dependent upon reactions with the sample and reference.

The ambient temperature is measured and controlled with an automatic pyrometer in conjunction with a programmer and proportional controller. The pyrometer is calibrated in place against the melting points of Au, Ni, Pt, Al_2O_3 , Mo, and Ir.

The melting points and phase equilibria have been measured for some actinide (Cm_2O_3) and lanthanide (Pm_2O_3 , Sm_2O_3) oxides. Reversible, reproducible reactions have been measured. For example, for Cm_2O_3 the following reactions were observed:

$1615 \pm 20^\circ\text{C}$	$\text{B} \rightleftharpoons \text{A}$
$2000 \pm 20^\circ\text{C}$	$\text{A} \rightleftharpoons \text{H}$
$2210 \pm 20^\circ\text{C}$	$\text{H} \rightleftharpoons \text{X}$
$2275 \pm 25^\circ\text{C}$	Melting

Similar phase transformations were obtained for Pm_2O_3 with a melting point of $2320 \pm 25^\circ\text{C}$.

The Pm_2O_3 - Sm_2O_3 binary system was also investigated, Figure 9. The lines separating the different regions should be two-phase regions; the lower and upper boundaries ($<30^\circ\text{C}$) could not be distinguished.

EXPERIMENTAL PROBLEMS

There are a number of problems associated with the measurement and evaluation of high temperature liquid properties of ceramic materials. These problems often require the use of novel and unusual methods if measurements are to be made. Perhaps the most difficult is the incompatibility of the sample with its container material. The elevated temperatures increase the rates of reactions and enhance the formation of new phases. In most instances, the choice of container material is narrow and often restricts the choice of experimental conditions, e.g., use of refractory metals require low oxygen pressures. Related to this problem is the difficulty in capsule fabrication. Closure is complicated by poor quality refractory metals and machining and welding techniques.

Vapor loss and sample decomposition always must be considered at these temperatures. However, these data for liquid ceramics are very limited. Loss of sample and compositional change may alter the properties of the liquid. It has been noted that both single crystal and polycrystalline oxides release significant amounts of absorbed gas on melting. Bubbles can form in the sample complicating property measurements and eventually the released gas,

Fig. 9. DTA results for Pm_2O_3 - Sm_2O_3 binary system.

coupled with a potential high sample vapor pressure, can cause excessive swelling of the sealed metal container.

If the vacuum-sealed capsule is measured in gas atmosphere, permeation of the gas through the container can occur. Choice of cover gas can minimize this problem.

Temperature measurement and calibration become increasingly more difficult as the temperature increases. It is impractical or impossible to measure the temperature of the liquid directly. Use of thermocouples is complicated by loss of electrical resistance of insulators, diffusion in thermocouple, reactions with the system and lack of calibrated emf's. This problem is partially solved by using self-supporting, bare refractory metal thermocouples in the hot zone of the furnace. Optical pyrometer measures are generally made simultaneously with thermocouple measurements as a consistent check of temperatures. Temperature calibration is difficult yet the system can be self calibrating for samples with known melting points since melting is easily observed.

High temperature references and standards are non-existent. Methods to evaluate the data often do not apply or have yet to be developed.

All these severe temperature conditions impose limitations which reduce the accuracy and precision of the data. However, it can be said, some data is better than no data at all with the hope that new and improved techniques can be developed which will improve the knowledge of the liquid state of ceramic materials.

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