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Thermal and Mechanical Properties of Nitrate Thermal Storage Salts in the Solid-Phase*

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Abstract

Implementation of molten salt compounds as the heat transfer fluid and energy storage medium provides specific benefits to energy collection and conversion. Nitrate salts have been identified as a strong candidate for energy transfer and storage and have been demonstrated for use in these applications over time. As nitrate salts have solidification temperatures above ambient, concern for recovery from salt freezing events has instigated efforts to understand and predict this behavior. Accurate information of salt property behavior in the solid-phase is necessary for understanding recovery from a freeze event as well as for phase change thermal energy storage applications. Thermal and mechanical properties for three representative salts (solar salt, HITEC salt, and a Na-K-Li-Ca nitrate salt; spanning the range of liquidus temperatures from approximately 90 – 240 °C), have been obtained. These properties include: specific heat, coefficient of thermal expansion, thermal conductivity, latent heat of fusion, compressive strength, tensile strength, Young's modulus and Poisson's ratio. Specific heat, thermal conductivity and latent heat of fusion were measured using differential scanning calorimetry. Temperature was not observed to have a significant effect on tensile strength using an indirect tensile test (Brazilian test). Peak stress and Young's modulus (both from unconfined compressive strength testing) were shown to decrease while Poisson's ratio increased with increasing temperature.

Keywords: nitrate salt, thermal storage, thermal and mechanical property, solid-phase

1. Introduction

Interest in raising the operating temperature of concentrating solar technologies and the incorporation of thermal storage has motivated studies on the implementation of molten salt as the system working fluid. Recently, salt has been considered for use in trough and linear Fresnel based solar collectors and has been shown to offer a reduction in levelized cost of energy as well as increased availability (Kearney *et al.*, 2003). Concerns regarding the use of molten salt in troughs are often related to issues with salt solidification and recovery from freeze events. Differences among salts used for heat transfer and storage are typically identified by a comparison of melting point, liquid-phase thermal properties and cost. However, the potential for a freeze event necessitates an understanding of salt thermal and mechanical properties in the solid-phase in order to characterize and mitigate possible detrimental effects during freezing and freeze event recovery. This includes stress imparted by the expanding salt. Initial modeling efforts of tube stress during freeze event recovery have been reported (Iverson *et al.*, 2011; Kolb *et al.*, 2010).

Additionally, significant attention has been given to using salts as a phase change storage media (using encapsulation and otherwise) (Goswami *et al.*, 1990; Herrmann and Kearney, 2002; Kenisarin, 2010; Steinmann and Tamme, 2008). Property changes as a function of temperature as well as an understanding of solid-phase behavior are critical in characterizing systems of this type. Salt volumetric changes and thermal transport are paramount in storage applications. For freeze event recovery and phase change thermal energy storage, reliable material property data are an integral part of model validation. Previous modeling efforts have assumed mechanical properties similar to published data on common salts. In this work, data is presented for measured thermal and mechanical properties of three salts to be used for heat transfer and storage in energy applications.

In an effort to span the typical range of melting point possibilities of thermal storage salts, three representative salts were selected for property testing: solar salt (60 wt% NaNO₃, 40 wt% KNO₃)(Bradshaw and Carling, 1987; Pacheco *et al.*, 1994), HITEC salt (40 wt% NaNO₂, 7 wt% NaNO₃, 53 wt% KNO₃)(Coastal Chemical Co., 2011) and a low melting point quaternary salt (42.3 wt% KNO₃, 39.4 wt% Ca(NO₃)₂, 12.1 wt% NaNO₃, 6.1 wt% LiNO₃)(Bradshaw *et al.*, 2009). Solar and quaternary salt compositions were mixed and melted for testing from individual components, whereas HITEC was acquired from Coastal Chemical Company (purity >99.6%). The solar salt and quaternary salt purities for the KNO₃, Ca(NO₃)₂, NaNO₃, and LiNO₃ constituents are 99.9%, 99.7%, >97% and 99.8% respectively. The quaternary salt has been studied because of its low melting point relative to the other two salts. However, to date, it has not been used in a concentrating solar power (CSP) facility. Table 1 lists the liquidus temperature (T_{liq}) for each salt along with a summary of the temperature ranges over which the tests were performed.

Samples of solar salt, HITEC salt and the low melting point quaternary salt were cast for solid-phase characterization tests to determine the following thermal and mechanical properties: specific heat, coefficient of thermal expansion (CTE), thermal conductivity, latent heat of fusion, unconfined compressive strength, indirect tensile strength, Young's modulus and Poisson's ratio. This work is presented as a summary and extension to the temperature dependent thermal and mechanical properties previously reported (Iverson *et al.*, 2010; Iverson *et al.*, 2011). Data collected in this study include force, temperature, and axial and radial displacements. Typically, these data are acquired using electronic transducers in which the electrical output is proportional to the change in the measured variable. In all property cases,

the constants of proportionality were determined through careful calibration using standards traceable to the National Institute for Standards and Technology. Both the thermal and mechanical properties were obtained following specific ASTM standards with deviations specified, where applicable.

2. Specific Heat

2.1 Sample Preparation and Test Procedure

Cylindrical pieces of each salt were cast in a PTFE tube of approximately 1 cm diameter with a silicone stopper and extracted for property testing. The sample container was at room temperature during pouring and were allowed to cool at room temperature. Sample sizes of roughly 100 mg were sectioned for specific heat measurement and placed in a hermetically sealed crucible to avoid water absorption.

The direct method of differential scanning calorimetry (DSC) was employed to measure the specific heat (c_p) of salt samples using a Mettler Toledo DSC 823 instrument (Mettler Toledo, 1998). The samples were subjected to a linear temperature-increase with no superimposed temperature fluctuations. The heat capacity can be determined simply from

$$c_p = \frac{\text{heat flow}}{\text{mass} \times \text{heating rate}} \quad (1)$$

where heat flow is a measured value from the DSC, the heating rate is set to 10 °C/min, and the mass is the weight of the sample. Test procedures called out in ASTM standard E1269 were utilized (ASTM-E1269-11, 2011).

For each salt, the sample temperature was increased from a temperature at or below room temperature up to at least 100 °C above the liquidus temperature. Each test was performed multiple times to ensure repeatability and the absence of water. Testing over a broad range of temperatures in this manner captures the specific heat observed throughout the solid-phase and provides an indication of the specific heat in the liquid phase at low temperatures for comparison. A 40 μ L aluminum crucible was used with samples under an argon atmosphere for testing with specific heat measured over the temperature range of interest using the direct method.

2.2 Test Results

Specific heat values as a function of temperature obtained using the direct DSC method described above are presented in Figure 1. Values were obtained from 10 °C through 400 °C for each salt. The measurement was repeated three times with nearly identical results. The average of the three runs for each salt is plotted in Figure 1a. The large initial spike for solar salt and HITEC salt is indicative of solid-solid phase transitions since neither mixture is a eutectic. In particular, the initial peak seen for 60/40 solar salt (60 wt% NaNO₃, 40 wt% KNO₃) corresponds to a peak observed in specific heat for KNO₃ as the 60/40 solar salt mixture contains more KNO₃ than the 54/46 eutectic mixture (Takahashi *et al.*, 1988). The large secondary spike for these salts indicates the solid-liquid phase change at the melting point. A slight increase is observed in the solid-phase specific heat with temperature whereas a flat profile is observed in the liquid-phase for the temperature ranges considered. The liquid-phase c_p value for solar salt at 1.50 kJ/kg·K (Table 2) is in excellent agreement with values reported in the literature (Rogers and

Janz, 1982). Values for specific heat at 25 °C, 20 °C below the liquidus temperature and 50 °C above the melt temperature for each salt is provided in Table 2 with errors of $\pm 7\%$.

Figure 1b illustrates specific heat values in the solid-phase up to about 100 °C for solar and HITEC salts and 30 °C for the quaternary salt. For this plot, the selected temperature range was limited so as to avoid solid-solid phase transition as well as the onset of melting. Data points from repeat measurements were included and a linear regression performed to indicate temperature dependence. The values for HITEC agree well with the single reported value of 1.34 kJ/kg·K, with no specific temperature provided (Coastal Chemical Co., 2011). The quaternary salt exhibits a very flat profile in the solid-phase up to about room temperature. However, in Figure 1a, a marked increase in c_p is observed below 50 °C, well below the liquidus temperature of 90 °C. In fact, this material exhibits a glasslike specific heat transition from solid to liquid without a large spike as observed in solar and HITEC salts. For this reason, the quaternary salt data points presented in Figure 1b, and used to calculate the solid-phase c_p , only include temperatures up to ~ 30 °C.

Figure 2 illustrates the melting process for the three salts (arranged in each picture in the following order: quaternary, HITEC and solar salt). Each image is at a different temperature with light-colored regions indicating opacity due to the presence of solid-phase components. At temperatures below the liquidus temperature of 90 °C, the quaternary salt exhibits phase transitions with glass-like behavior, indicative of early melting.

3. Coefficient of Thermal Expansion

3.1 CTE Sample Preparation

Cylindrical samples (with diameter of approximately 5.08 cm and cut to length) were cast in a PTFE tube with silicone stopper and extracted for property testing (Figure 3a-b). Molten salt was poured so as to fill the PTFE tube contiguously and avoid layer formation in the salt. The sample container was at room temperature during pouring and were allowed to cool at room temperature. Cratering near the top of the samples (due to phase-dependent density change) and void formation in the salt-core interior were avoided by cross sectioning and identifying unaffected regions of the solidified salt (Figure 3c). Solar salt, which has a large volume increase during phase change from solid to liquid (4.6%), had the most pronounced cratering (Bradshaw and Carling, 1987; Pacheco *et al.*, 1994). The quaternary salt had no visible cratering.

All CTE samples of were cut using a diamond wire saw to nominally 4.7 cm in length. Solar salt and HITEC samples were turned on a lathe to 2.3 cm in diameter. Quaternary salt samples proved to be very difficult to machine given their glasslike behavior; therefore, samples were cast to a diameter of 1.5 cm before being cut to length. The estimated porosity is less than about 5% for all salt types, as computed from the measured dimensional density divided by the sum of the constituent theoretical densities. Two polycrystalline samples of each salt material with undetermined grain size were sent to Harrop Industries, Inc. for CTE measurement.

3.2 CTE Test Procedure and Results

Salt samples were loaded into a temperature controlled oven with a long push rod and heated at 3 °C/min. Each sample was heated and then cooled back to its starting temperature. The target high temperature was approximately 90% of the liquidus temperature. Expansion and contraction of the salt was recorded using linear displacement transducers relative to the

starting point throughout the test procedure. The test procedure follows ASTM E228 (ASTM-E228-11, 2011).

Figure 4 and 5 illustrate the percent elongation (relative to the initial length at room temperature) as a function of temperature for all three salt types. A repeat experiment was performed for each salt type yielding similar results; values for all CTE experiments are provided in Table 3. Solid lines in Figure 4 and 5 represent actual data collected (at 0.1-0.2 °C increments) while the dashed and dotted lines represent the least-squares best fit (linear regression) over the heating and cooling regions. The slopes of these curve fits correspond with the best-fit values in Table 3. Averages of the two test samples for each material over the heating and cooling regions are provided in the “Avg” column of Table 3. For samples of solar salt and HITEC, there appears to be continued expansion of the materials when the temperature is decreasing. This effect may indicate that there was some thermal inertia due to a possible non-uniform temperature throughout the sample. The values of CTE for solar salt and HITEC are similar for both heating and cooling (Table 3). Both salts exhibit rather linear thermal expansion upon heating and both expand slightly for the first 10-20 °C of cooling. A single reported value for the thermal expansion of solid-phase HITEC salt has been reported as $51.3 \times 10^{-6} \text{ }^{\circ}\text{C}^{-1}$ (Coastal Chemical Co., 2011). This value agrees well with the CTE values measured upon heating (see Table 3).

For the purpose of comparison, the CTE of Halite (NaCl) is presented at the bottom of Table 3. The first value of $40 \times 10^{-6} \text{ }^{\circ}\text{C}^{-1}$ was selected at a temperature near the midrange (~75 °C) of test temperatures for solar salt and HITEC (Drebushchak and Turkin, 2001). The second value given for Halite of $45.0 \times 10^{-6} \text{ }^{\circ}\text{C}^{-1}$ was defined as the coefficient of linear thermal expansion and therefore implies temperature independence (Callahan and DeVries, 1994). CTE for solar salt and HITEC are more than 30% higher than that for naturally occurring polycrystalline Halite. As an additional comparison, the coefficient of thermal expansion for steels ranges from $10\text{-}20 \times 10^{-6} \text{ }^{\circ}\text{C}^{-1}$ (MatWeb, 2011). This CTE difference between salts and steel can be critical in thermomechanical modeling.

The behavior of the quaternary salt (Figure 5) is markedly different from that for solar salt and HITEC (Figure 4). A small length change was observed upon heating to about 50 °C. From 50 °C to 90% of the liquidus temperature, the quaternary salt exhibited contraction. The contraction is a result of the solid to liquid phase change that begins to take place at temperatures below 65°C (as seen in Figure 2). The CTE was calculated within the linear region (28-42 °C for both the heating and cooling cycles) before the expected onset of the solid to liquid phase change (as shown in Figure 5). Calculating the CTE within this region best represents the material behavior in the solid-phase. The temperature ranges and best-fit values listed in Table 3 are based on this limited range. For the starting temperature up to 42 °C and from 42 °C down to room temperature, the CTE of the quaternary salt averages $56.4 \times 10^{-6} \text{ }^{\circ}\text{C}^{-1}$ and $29.2 \times 10^{-6} \text{ }^{\circ}\text{C}^{-1}$, respectively.

4. Thermal Conductivity

4.1 Thermal Conductivity Sample Preparation

Similar to the CTE samples, cylindrical pieces of each salt were cast in a PTFE tube with silicone stopper and extracted for property testing. Molten salt was poured so as to fill the PTFE tube contiguously and avoid layer formation in the salt. The sample container was at room temperature during pouring and were allowed to cool at room temperature. Cratering was

again avoided by cross sectioning and identifying unaffected regions. Initial cutting of all thermal conductivity samples was performed using a lathe to achieve a prescribed diameter (5.84 mm) and then cut using a diamond wire saw to small segments a few mm in length. These segments were then sanded to achieve thickness of 1.5-3.0 mm. Special care was taken to ensure that the sides of small disks were perpendicular to the flat ends of the cylinder. Samples were stored in a desiccant chamber to prevent water absorption. Owing to the glasslike nature of the quaternary salt, machining parts to a diameter of 5.84 mm was problematic. This precluded the quaternary salts from being evaluated using the methods employed here for thermal conductivity.

4.2 Thermal Conductivity Test Procedure

Following a procedure established by Riesen (Riesen, 2005), DSC was used to establish heat flow through a sample salt disk to an aluminum crucible containing the low melting-point metal gallium ($T_m = 29.76^\circ\text{C}$, see Figure 6). As gallium is the reference material in all test cases, the temperature of the conductivity measurements was limited to 30°C . Indium was used to calibrate the heat flow of the DSC. Interfaces between the salt disk, heated surface and crucible were filled with a small amount heat transfer oil to maintain reproducibility.

The total resistance to heat flow through this stack is the proportionality constant between the thermal power (q) and temperature difference (ΔT). For the oil interface resistances (R_{int}) and sample resistance (R_s) between the metal and heated surface, we obtain

$$q = \frac{\Delta T}{\Sigma R} = \frac{T_h - T_m}{R_{int} + R_s + R_{int}} \quad (2)$$

The melt temperature of the metal (T_m) is known and the heated surface temperature ($T_h = 31$ to 33°C) and q are obtained during the DSC measurement. The resistance due to the oil at both interfaces and across test samples can be assumed to be the same since the same sample cross section is used. Thus, we define

$$R_{t,int} = R_{int} + R_{int} \quad (3)$$

where the total interface resistance ($R_{t,int}$) can be determined by performing multiple measurements on similar samples. If $R_{t,int} \ll R_s$, then $R_{t,int}$ can be neglected.

Assuming a small $R_{t,int}$, one-dimensional heat flow, steady state, and no internal generation, the heat flow is defined by

$$q = \frac{kA}{L}(T_h - T_m) \quad (4)$$

where L is the thickness of the sample, k is the sample thermal conductivity, and A is the cross sectional area. Equation 4 can be solved for the conductivity of the material directly from a single melting curve, assuming $R_{t,int}$ is small. When $R_{t,int}$ is not negligible, we can define the slope of the linear side of the melting peak (see also Figure 7a) as

$$S = \frac{q(t) - q_{onset}}{T_h(t) - T_{onset}} = \frac{1}{R_{t,int} + L/kA} \quad (5)$$

where q_{onset} and T_{onset} are the heat flow and melt temperature of metal at the onset of melting. When two samples of the same material and different thicknesses are measured, the thermal conductivity of the sample can be obtained by

$$k = \frac{\Delta L}{A \left(\frac{1}{S_2} - \frac{1}{S_1} \right)} \quad (6)$$

When several samples of different heights are used, the thermal conductivity and $R_{t,int}$ can be determined by rearranging equation 5 and using a linear regression to obtain k , as follows

$$\frac{1}{S} = \frac{L}{kA} + R_{t,int} \quad (7)$$

4.3 Thermal Conductivity Test Results

As a validation of the procedure outlined for thermal conductivity above, measurements were made on PTFE and compared to literature values and previous DSC based measurements. Using a direct approach following equation 4 (neglecting interface resistance) and averaging over the linear range of the heat flow vs. temperature curve, we obtained a thermal conductivity value of 0.206 W/m·K for PTFE (error of 17.6%). By using the linear regression approach following equation 7 on multiple measurements of PTFE with varying thicknesses, we obtained 0.247 W/m·K (error of 1.2%). These values agree well with an accepted literature value of 0.25 W/m·K and is an improvement over the 0.181 W/m·K measurement reported in Mettler Toledo's UserCom (Riesen, 2005) thus lending confidence to the measurement technique.

Figure 7a illustrates an example heat flow curve as a function of temperature for a solar salt sample of thickness 2.5 mm. Here the onset of melting of the metal (Figure 6) is observed at approximately 30.5 °C. The linear portion of this curve, immediately following the onset of melt is used to determine the slope parameter, S (equation 5), for each measurement taken. The inverse of this slope from the linear region of each sample is then plotted as a function of the geometric ratio L/A as in Figure 7b and Figure 8. Samples were prepared in four thicknesses; multiple stacked samples provided the upper range of material thicknesses as indicated in the figures. Thermal conductivity is obtained in two ways from the data in these figures. First, thermal conductivity was measured directly (k_{direct}) following equation 4 and averaged over all samples. Second, a linear regression was performed to represent the measurement data of Figure 7b and Figure 8 where the inverse of the slope of the regression model yields thermal conductivity of the material ($k_{regression}$). Measured thermal conductivity values obtained in these fashions are listed in Table 4 along with standard deviation and R^2 values to indicate variability.

Although made of different compositions, solar salt and HITEC demonstrate similar thermal conductivity values. Previous reporting indicates that the thermal conductivity of HITEC in the liquid phase appears to approach 0.44 W/m·K as the temperature decreases towards solidification (Coastal Chemical Co., 2011). However, to the author's knowledge, information for these salt mixtures below the solidification temperature has not been presented previously. As a comparison, thermal conductivity values for NaNO_3 have been reported and range from approximately 0.7-0.9 W/m·K (Bauer *et al.*, 2012).

5. Heat of Fusion

5.1 Heat of Fusion Sample Preparation

Rough cast samples for use in measuring thermal conductivity were also used for heat of fusion measurements. Samples (10-30 mg) were fractured from a larger sample and then placed in hermetically sealed aluminum pans (40 μ L). To avoid water absorption and deliquescence (as nitrate salts are hygroscopic), samples were handled after casting in a glove box under an inert atmosphere of argon. The aluminum pan lid was pierced immediately before acquiring data in the DSC. At least three measurements on different samples were performed and averaged.

5.2 Heat of Fusion Test Procedure and Results

Previous work indicated that the heat of fusion for mixtures of nitrate salts does not follow ideal mixing behavior, making it necessary to measure the apparent heat of fusion for different mixtures (Cordaro *et al.*, 2011). DSC was utilized to measure the apparent heat of melting/fusion (ΔH). Similar to the ASME E968 standard (ASTM-E968-02, 2008), a Mettler-Toledo DSC 823e equipped with an auto-sampler was calibrated using the melting point and heat of fusion for Indium and melting point of Zinc. A heating rate of 10 $^{\circ}$ C/min was used for all measurements.

To test the validity of this method, the heat of fusion for sodium nitrate (NaNO_3) was measured and compared to literature values with good agreement (see Table 5). Table 5 also lists the heat of fusion data for all salt materials along with values reported in the literature, where available. Heating curves for NaNO_3 show a narrow endothermic signal (Figure 9). On the contrary, the quaternary salt displays non-eutectic behavior as evident by the broad endothermic curve. As an off-eutectic mixture with glass-like behavior, the quaternary salt displays a broad endothermic heat flow between 30 and 90 $^{\circ}$ C. Full melting does not occur until 90 $^{\circ}$ C, as determined by using a conventional melting point apparatus (Figure 2). Because of the broad range over which the sample melts and the glass-like nature of calcium containing salts, the heat of fusion is difficult to accurately measure. The value of 15.2 [J/g] reported here, however, reflects the glassy nature of calcium containing salts, which can have an unexpectedly low apparent heat of fusion due to amorphous packing of the ions in the solid state (Angell and Tucker, 1974). Heat of fusion data is available in the literature for solar salt and HITEC salt; this data is also included in Table 5.

6. Tensile Strength

6.1 Tensile Strength Sample Preparation

Similar to CTE sample preparation, cylindrical samples (with diameter of approximately 5.08 cm and cut to length) were cast in a PTFE tube with silicone stopper and extracted for property testing (see Figure 3a-b). Molten salt was poured so as to fill the PTFE tube contiguously and avoid layer formation in the salt. The samples were cooled at room temperature. Cratering near the top of the samples (due to phase-dependent density change) and void formation in the salt-core interior were avoided by cross-sectioning and identifying unaffected regions of the solidified salt, as before (Figure 3c).

The indirect tensile test (also known as a Brazilian disc tension test) commonly used on rock and concrete, was employed to generate a nearly uniform tensile stress distribution normal to the

compression-loaded vertical plane to obtain tensile strength data. Specimens used for tensile strength determinations were sawn from cast cylinders to produce L/D ratios of 0.5; the ends of the specimen were not ground. For indirect tension specimens, the sides were left undressed to avoid damage that could occur in standard machining operations. Indirect tension specimens were prepared following the general outline given by ASTM Standard D3967 (ASTM-D3967-08, 2008).

At the completion of the specimen preparation step, the density of each test specimen was calculated from the measured mass divided by the sample volume calculated from the specimen dimensions, assuming each specimen was a perfect cylinder. Each sample was labeled as follows: project (SS) – type of test (UC = unconfined compression test, BZ = Brazilian tensile test) – nominal test temperature (in °C) – sample material (A = solar salt, B = HITEC, C = quaternary salt) – sample number. For example, SS-BZ21-B02 indicates a Brazilian tensile test at room temperature with HITEC salt on sample 2.

6.2 Tensile Strength Test Method

Tensile strength was determined using ASTM D3967 (ASTM-D3967-08, 2008) with the following two exceptions: loading rate and number of specimens. An initial trial test concluded that a higher loading rate of 50.8 $\mu\text{m}/\text{sec}$ was required. At this loading rate, samples failed in approximately 10 seconds; the minimum recommended time for sample loading in ASTM D3967 is 1 minute. The higher loading rate was necessary to counter sample creep at elevated temperatures so as to induce sample failure before large creep deformations occur. ASTM D3967 states that a minimum of ten samples should be tested to produce a meaningful average value unless reproducibility is good. The coefficient of variation (standard deviation/mean) for solar, HITEC and quaternary salt tensile strength measurements was 38%, 17% and 34%, respectively. Also, the number of samples tested was seven, six and four respectively.

The splitting tensile strength test (or Brazilian test) is an indirect method for determining tensile strength because compressive stresses are applied in one direction that, in turn, induce tensile stresses in an orthogonal direction. Tensile strength can also be determined using a direct-pull, uniaxial test configuration, but such a method is more difficult to perform and more expensive when compared to the indirect method. Therefore, tensile strengths measured in this study made use of the indirect method and thus will be referred to as indirect tensile strengths. In the splitting tensile strength test, typically a rock disk, having a L/D = 0.5, is diametrically loaded between rigid platens (with bearing strips) until failure occurs. Because of the arc shape of the disks, the loading is assumed to be along a line rather than distributed over some area. When the compressive line loading induces tensile stresses that reach the tensile strength of the material, the material fails typically by means of a vertical fracture parallel to the compressive line load (Figure 10a,b). The splitting tensile strength is calculated as,

$$\sigma_t = 2P/\pi LD \quad (8)$$

where σ_t is the splitting tensile strength [Pa], P is the maximum applied load [N], L is specimen thickness [m], and D is the specimen diameter [m].

6.3 Indirect Tensile Test Results

Table A.1 summarizes the results for all indirect tension tests. Seventeen tests were completed successfully: seven for solar salt, six for HITEC, and four for the quaternary salt. Early in the testing series, samples were placed between cardboard loading strips. Because of the high deformation observed in the material, the cardboard loading strips were removed to aid in

sample failure. Subsequent samples were taped around the circumference with two layers of masking tape. The masking tape served two purposes in that it acted as a very thin loading strip and also kept the sample intact after failure. Sample SS-BZ127-A05 did not fail (as noted in Table A.1) so the tensile strength was determined from the load that was observed coincident with the greatest deformation.

Large deformations were observed while testing specimen SS-BZ127-A05 at 127 °C so it was decided to test two samples at 78 °C. In addition, one test at 63°C was completed on solar salt to provide data at an intermediate temperature. Many indirect tension test samples, especially those tested at elevated temperature, exhibited large deformation before failure. As a result, the curved surfaces of the test samples in contact with the loading platens flattened such that the applied line load transitioned to a load distributed over the flattened area. Figure 10b shows an example where the load is distributed over a flattened area before failure. Distributed loads with an arc of 15° or less give similar tensile strength values as those derived from line loads and fail with similar diametral fractures provided the load is corrected to account for the contrast between line loading and distributed loading (Jaeger and Cook, 1979). Table A.1 lists two columns for tensile strength, one in which the line load assumption is valid (i.e., no observed flattening of the samples) and one in which the line load assumption is invalid (significant flattening is observed). This latter tensile strength is defined in Table A.1 as corrected tensile strength. The flattened lengths (distance between the loading arrows shown in Figure 10b) for those tests in which the line load assumption is invalid were measured post-test and are also given in Table A.1. The corrected failure load is given by (Jaeger and Cook, 1979),

$$W = 2par \quad (9)$$

where W is the corrected load at failure [N], p is the load per unit length [N/m], α is half the angle subtended by the flattened region [rad] and r is the specimen radius [m]. Using the calculated corrected load, the corrected indirect tensile strength ($\sigma_{t,c}$) can be calculated by replacing the maximum applied load (P) in equation 8 with the corrected failure load (W , equation 9).

Figure 11a plots indirect tensile strength versus temperature for all salt types. The variability in tensile strength at all temperatures for each salt type is large but not atypical of this type of index test measurement. For a given test temperature, the weakest sample from repeated tests is approximately 50% of the strength of the strongest sample for the same salt type, a trend also seen in natural rock salts. All salt types are much stronger than naturally occurring rock salts. As a reference, a typical upper range of indirect tensile strength for rock salts is approximately 2 MPa (300 psi) based on several locations (Hansen *et al.*, 1981). The rock salt locations represent solar salts well because, like naturally occurring rock salts, solar salts are aggregates and polycrystalline in nature. A strong dependence of indirect tensile strength on temperature is not observed over the temperature ranges tested for this small data set. The average indirect tensile strength for solar salt, HITEC and the quaternary salt is 4.13, 3.60, and 3.83 MPa, respectively.

7. Compressive Strength

7.1 Compressive Strength Sample Preparation

Sample preparation followed that outlined for tensile testing. Samples were cut to length using a diamond wire saw. Unconfined compressive strength (UCS) samples were cut from near the bottom of each casting while maintaining an approximate 2/1 length-to-diameter ratio (L/D). Next, the specimens were mounted in a surface grinder where the ends of the specimens were carefully ground flat and parallel to within a tolerance of 0.0254 mm (0.001 inches). Preparation of the UCS samples followed the general outline given by ASTM Standard D4543 (ASTM-D4543, 1995). One sample of each salt type was turned down to a smaller diameter using a lathe. This operation was done to ensure the UCS could be obtained within the capacity of the loading frame used for elevated temperature testing. All other UCS testing was performed on as-received sample diameters.

7.2 Unconfined Compressive Strength Test Method

The UCS test was used to determine compressive strength. Unload and reload cycles performed during this test were used to obtain Young's modulus and Poisson's ratio. UCS tests were conducted by applying compressive loads to the ends of right-circular cylindrical salt specimens ($L/D \approx 2$) in axial strain control until failure occurred as denoted by a drop in the axial load. The UCS is then calculated as the peak or maximum compressive force applied to the sample ends divided by the sample cross-sectional area. Electronic transducers were mounted on the test samples to measure axial and lateral sample deformations (Figure 10c illustrates a typical instrumented UCS sample and shows the axial and lateral linear variable differential transformer (LVDT) displacement transducers). Data acquired from the LVDTs together with axial stress were used to determine elastic properties such as Young's modulus and Poisson's ratio. During elevated temperature testing, samples were placed inside an environmental chamber (Figure 10c) that controlled the temperature to within $\pm 1^\circ\text{C}$.

For some room-temperature tests, the axial load required to fail the samples exceeded the 22,000 lbs. capacity of the two-column reaction frame used with the environmental chamber test stand. As a result, subsequent room-temperature tests were conducted on a larger capacity (220,000 lbs.) four-column load frame. Each load frame is equipped with a load cell placed in line with the sample to measure the axial force applied to the sample ends. The first UCS test was conducted at an axial strain rate of 1×10^{-4} /sec. However, sample creep was observed during the unload/reload loops performed for elastic property determination so all subsequent tests were performed using an axial strain rate of approximately 1×10^{-3} /sec to mitigate sample creep and ensure accurate determination of material properties.

7.2 Unconfined Compressive Strength Results

The UCS tests of all three salts were determined by standard compression tests of right circular cylinders as described previously. Seventeen tests were completed: eight for solar salt, five for HITEC, and four for the quaternary salt. Table A.2 lists the peak stress observed in each test along with the density for all samples. Although peak stress is generally synonymous with UCS, there were a few tests in which UCS could not be obtained because loading needed to be stopped before a stress drop (failure) was observed given the large sample deformations that occurred. In these cases, the peak stress was reported although this stress may be somewhat lower than the actual UCS.

Figure 11b plots peak stress versus temperature for all UCS samples. Linear fits using a least squares regression are given for each salt type comparing UCS (or peak stress) to temperature. For all salt types, UCS decreases as temperature increases. Scatter in the data is observed and is expected for this type of index test and material. Variations in void concentration will impact sample density. However, there does not appear to be an obvious trend between density and UCS in these results. If a correlation does exist for density and other mechanical properties, it is likely overshadowed by temperature variations and the relative small range of densities observed for the test specimens.

8. Young's Modulus and Poisson's Ratio

Young's modulus (E) and Poisson's ratio (ν) were determined from true axial stress versus true strain data using least squares linear regression. Young's modulus is defined as,

$$E = \Delta\sigma_a / \Delta\varepsilon_a \quad (10)$$

where $\Delta\sigma_a$ is the change in true axial stress and $\Delta\varepsilon_a$ is the corresponding change in true axial strain. Poisson's ratio is defined as,

$$\nu = E / (\Delta\sigma_a / \Delta\varepsilon_l) \quad (11)$$

where $\Delta\varepsilon_l$ is the change in true lateral strain corresponding to the change in true axial stress. An example stress versus strain plot is shown in Figure 12a. Figure 12b shows a zoomed in region of Figure 12a and illustrates the linear regions used in calculating E and ν in the unload/reload cycles.

Table A.3 shows all calculated E and ν values. The column labeled 'stress at unload' is the highest axial stress in the interval where E and ν were determined. The stress at unload is only given for elastic properties calculated from unload/reload cycles. For cases where the sample had reached dilation (the stress at which volumetric strain attains its most positive value, sample compaction), Poisson's ratio is not reported. When salt dilates, micro-cracks form and the material begins to rapidly expand in the radial direction leading to negative volumetric strains.

Figure 13 illustrates the relationship of E and ν with temperature for all salt types. Least squares linear regression fits are presented for the E and ν trends with temperature. All elastic properties are plotted including initial E and ν values as well as those calculated from the unload/reload cycles. All three salts exhibit a decrease in E with increasing temperature likely due to material softening. Poisson's ratio tends to increase with increasing temperature. For the quaternary salt, a possible outlier in the data has been excluded from the trend line fit. The possible outlier is plotted for completeness.

9. Conclusion

Thermal and mechanical properties of three representative salts for use in thermal storage systems have been evaluated as a function of temperature. The nitrate salts (solar, HITEC, and quaternary salts) span a wide range of liquidus temperatures from 90 – 238 °C. Measured values for coefficient of thermal expansion were obtained using linear displacement transducers with sample temperatures ramped to approximately 90% of the melting temperature. Thermal conductivity, specific heat, and the apparent heat of fusion were obtained using a differential scanning calorimeter. As an off-eutectic mixture, thermophysical properties of the quaternary

salt were not as well behaved as solar salt or HITEC. Mechanical test results indicate that as test temperature increases, unconfined compressive strength and Young's modulus decrease for all salt types whereas Poisson's ratio tends to increase. Temperature was not observed to have a significant effect on tensile strength using an indirect tensile test (Brazilian test). The variability in measured indirect tensile strength is large, but not atypical for this index test. The average tensile strength for all salt types tested is substantially higher than the upper range of tensile strengths for naturally occurring rock salts. With the solid-phase behavior of common salts used in heat transfer and storage of energy systems now evaluated, thermomechanical modeling of freeze recovery strategies and phase change storage is enabled.

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Table 1. Approximate temperature ranges for thermal and mechanical property testing of each material.

Property	Solar Salt $T_{liq,A} = 238\text{ }^{\circ}\text{C}$	HITEC $T_{liq,B} = 142\text{ }^{\circ}\text{C}$	Quaternary $T_{liq,C} = 90\text{ }^{\circ}\text{C}$
Specific Heat	10 - 400 $^{\circ}\text{C}$	10 - 400 $^{\circ}\text{C}$	10 - 400 $^{\circ}\text{C}$
CTE	30 - 200 $^{\circ}\text{C}$	30 - 120 $^{\circ}\text{C}$	30 - 75 $^{\circ}\text{C}$
Thermal Conductivity	30 - 35 $^{\circ}\text{C}$	30 - 35 $^{\circ}\text{C}$	n/a
Unconfined Compressive Strength	21 - 127 $^{\circ}\text{C}$	21 - 84 $^{\circ}\text{C}$	21 - 39 $^{\circ}\text{C}$
Tensile Strength	24 - 127 $^{\circ}\text{C}$	28 - 84 $^{\circ}\text{C}$	27 - 40 $^{\circ}\text{C}$
Young's Modulus [*]	21 - 127 $^{\circ}\text{C}$	21 - 84 $^{\circ}\text{C}$	21 - 39 $^{\circ}\text{C}$
Poisson's Ratio [*]	21 - 127 $^{\circ}\text{C}$	21 - 84 $^{\circ}\text{C}$	21 - 39 $^{\circ}\text{C}$

^{*} Determined from unload/reload cycles

Table 2. Specific heat values at specified temperatures for solar salt, HITEC and quaternary salts. Values in the solid-phase were obtained from the linear regression equations in Figure 1b; liquid values were obtained from an average of three repeat measurements at the specified temperatures.

Temperature	Phase	c_p [kJ/kg·K] (error $\pm 7\%$)		
		Solar Salt	HITEC	Quaternary
25 $^{\circ}\text{C}$	solid	1.05	1.09	1.03
$T_{liq} - 20\text{ }^{\circ}\text{C}$	solid	1.85	2.82 [*]	n/a
$T_{liq} + 50\text{ }^{\circ}\text{C}$	liquid	1.50	1.57	1.58

^{*} Value obtained near solid-solid phase transition

Table 3. Coefficient of thermal expansion values over specified temperature ranges.

Salt type	Sample	Heat/cool range [°C]	$\alpha / 10^{-6} \text{ }^{\circ}\text{C}^{-1}$	
			Best fit	Avg
Solar salt	1A	Heat 30.1 – 193.3	54.2	Heat
		Cool 193.3 – 30.4	71.4	54.7
	2A	Heat 30.0 – 199.2	55.2	Cool
		Cool 199.2 – 30.4	69.8	70.6
HITEC	3B	Heat 30.0 – 121.6	54.9	Heat
		Cool 121.6 – 30.4	84.9	55.8
	4B	Heat 30.0 – 120.2	56.7	Cool
		Cool 120.2 – 30.4	83.7	84.3
Quaternary	5C	Heat 28.0 – 42.0	46.2	Heat
		Cool 42.0 – 28.5	33.6	56.4
	6C	Heat 25.0 – 42.0	66.5	Cool
		Cool 42.0 – 35.7	24.8	29.2
Halite (NaCl)	N/A	Heat (~75 °C)	~ 40 (Drebushchak and Turkin, 2001)	
		unknown	45.0 (Callahan and DeVries, 1994)	

Table 4. Measured thermal conductivity at approximately 30 °C for solar salt and HITEC using direct and linear regression approaches.

	Solar Salt	HITEC
k_{direct} [W/m·K]	0.76	0.74
Standard deviation	0.12	0.10
$k_{regression}$ [W/m·K]	0.79	0.70
R ² value of regression	0.96	0.93

Table 5. Salt heats of fusion (measured and literature values) with the inclusion of NaNO₃ as a baseline for comparison.

Salt	ΔH [J/g]	Literature value [J/g]
NaNO ₃	168.1 ($\pm$ 4.5)	173.4 (Berg <i>et al.</i> , 2006)
		181.7 (Rogers and Janz, 1982)
		173.3 (Cordaro <i>et al.</i> , 2011)
		173.0 (Takahashi <i>et al.</i> , 1988)
Solar Salt	-	109.8 (Rogers and Janz, 1982)
		133 (Coastal Chemical Co., 2011)
		161 (Pacheco <i>et al.</i> , 1994)
HITEC	-	81 (Coastal Chemical Co., 2011)
Quaternary	15.2 ($\pm$ 0.5)	n/a

(a)

(b)

Figure 1. Specific heat as a function of temperature for solar, HITEC and quaternary salts (a) from ambient through 400 °C (average of three tests) and (b) from ambient to just below the onset of melting or solid-solid phase transitions (for multiple tests with 5% of data points shown).

Figure 2. Images illustrating light intensity passing through the salts (in order from left to right of increasing melt temperature, i.e., quaternary, HITEC, and solar salt) for six temperatures ranging from room temperature to 245 °C. Lighter colors indicate less light passage due to the presence of the solid-phase.

(a)

(b)

(c)

Figure 3. (a) Prepared salt sample, (b) heating system and fixtures, (c) void formation in solar salt.

(a)

(b)

Figure 4. Percent length change as a function of temperature for (a) solar salt sample 1A, (b) HITEC salt sample 3B. Data points are taken at 0.1-0.2 °C increments with error values of <0.05% in length change and ± 1 °C in temperature. Trend lines are generated using all data points from the heating or cooling regions.

Figure 5. Percent length change as a function of temperature for quaternary salt sample 5C. Data points are taken at 0.1-0.2 °C increments with error values of <0.05% in length change and ± 1 °C in temperature. Trend lines are based on the data points collected only for the temperature range from 28.0-42.0 °C for heating and 28.5-42.0 °C for cooling as indicated by selected data points on the figure.

Figure 6. Test configuration for thermal conductivity using DSC.

(a)

(b)

Figure 7. (a) Example heat flow vs. temperature curve for solar salt sample of thickness 2.5 mm. (b) Measurement data for solar salt samples of 4 different thicknesses and a combination of multiple stacked samples following equation 7. The inverse of the slope of the linear regression trend line yields the regression-based thermal conductivity of the material.

Figure 8. Measured data for HITEC samples of 4 different thicknesses and a combination of multiple stacked samples following equation 7. The inverse of the slope of the linear regression trend line yields the regression-based thermal conductivity of the material.

Figure 9. Representative DSC heating curves for NaNO_3 and quaternary salts illustrating the latent heat behavior during phase change.

Figure 10. (a) Post indirect tensile strength test images of samples SS-BZ63-B03 and SS-BZ21-B01. Note the horizontal diametric crack along which the samples break when deformed. (b) Example of load distributed over an area for an elevated temperature indirect tensile test. (c) UCS test instrumented with axial and lateral displacement transducers in environmental chamber.

Figure 11. (a) Indirect tensile strength showing values relative to the typical upper range from naturally occurring rock salts (Hansen *et al.*, 1981) and (b) peak stress from UCS testing vs. temperature for solar salt, HITEC salt and a quaternary salt (see also Table A.1 and Table A.2).

(a)

(b)

Figure 12. True stress versus true strain for sample SS-UC27-A08 (a) for multiple unload/reload cycles and a (b) zoomed in view including linear regions sampled for E and ν (variance in E and ν values provided in the inset for multiple runs).

(a)

(b)

Figure 13. (a) Young's modulus and (b) Poisson's ratio as a function of temperature (see also data in Table A.3).

Appendix A

Table A.1. Indirect tension test results.

Sample	Test Temp. T_t [°C]	Homologous Temp. T_t/T_m [%]	Density [g/cc]	Indirect Tensile Strength [MPa]	Flattened Length [mm]	Corrected Tensile Strength*** [MPa]
*SS-BZ27-A01	27	61	2.16	N/A	0	N/A
*SS-BZ27-A02	27	61	2.17	6.62	0	6.62
SS-BZ27-A04	27	61	-	3.37	0	3.37
SS-BZ24-A08	24	60	2.13	3.85	0	3.85
SS-BZ63-A03	63	68	-	5.54	0	5.54
SS-BZ78-A06	78	71	-	2.64	7.3	2.57
SS-BZ78-A07	78	71	2.17	4.18	4.9	4.13
**SS-BZ127-A05	127	81	2.15	3.05	11.9	2.84
SS-BZ28-B01	28	73	2.11	3.12	0	3.12
SS-BZ29-B02	29	73	2.11	3.77	0	3.77
SS-BZ63-B03	63	81	2.09	3.24	4.2	3.20
SS-BZ63-B05	63	81	2.11	3.16	3.8	3.13
SS-BZ84-B04	84	86	2.08	4.86	7.6	4.68
SS-BZ84-B06	84	86	2.11	3.79	6.9	3.68
*SS-BZ27-C01	27	83	2.15	3.59	0	3.59
SS-BZ27-C03	27	83	-	3.05	0	3.05
SS-BZ39-C02	40	86	2.18	5.74	0	5.74
SS-BZ39-C04	39	86	2.19	2.94	0	2.94

N/A = load control of sample lost during test
 *Cardboard loading strips

**Sample did not break

***Jaeger and Cook, Figure 6.11.1c (Jaeger and Cook, 1979)

Table A.2. Unconfined compressive strength test results: density and peak stress.

Sample	Length [cm]	Diameter [cm]	Weight [g]	Density [g/cc]	Test Temp. T_t [°C]	Homologous Temp. T_v/T_m [%]	Peak Stress [MPa]
*SS-UC21-A05	9.26	4.36	299.8	2.17	21	60	129.62
*SS-UC21-A06	9.59	4.36	310.3	2.17	21	60	163.16
SS-UC23-A07	9.69	4.37	314.8	2.17	23	60	180.32
SS-UC27-A08	8.96	4.35	289.5	2.18	27	61	185.75
SS-UC78-A02	9.13	4.38	294.7	2.15	78	71	63.65
SS-UC78-A04	8.83	3.97	235.0	2.15	78	71	42.22
SS-UC127-A01	8.88	4.37	288.6	2.17	127	81	33.93
SS-UC127-A03	9.18	4.35	297.6	2.18	127	81	8.28
SS-UC21-B02	10.54	4.95	423.7	2.09	21	71	79.92
SS-UC57-B03	10.40	4.98	427.8	2.11	57	80	47.48
SS-UC63-B04	8.96	4.37	284.3	2.12	63	81	54.85
SS-UC63-B05	8.94	4.04	242.4	2.11	63	81	46.08
SS-UC84-B06	9.24	4.36	294.2	2.13	84	86	31.87
SS-UC21-C01	10.57	5.00	N/A	N/A	21	81	51.11
*SS-UC21-C04	8.95	4.21	276.6	2.22	21	81	29.28
SS-UC39-C02	10.42	5.01	449.7	2.19	39	86	3.70
SS-UC39-C03	10.34	5.04	448.7	2.18	39	86	6.68

N/A = Weight and density not determined pre-test

*Peak stress defined in terms of engineering stress; all others in terms of true stress

Table A.3. UCS test results: Young's modulus and Poisson's ratio.

Sample	Test Temp. T_t [°C]	Homologous Temp. T_i/T_m [%]	Young's Modulus, E [GPa]	Stress at unload* [MPa]	Poisson's Ratio, ν [-]
SS-UC21-A05	21	60	N/A		N/A
SS-UC21-A06	21	60	N/A		N/A
SS-UC23-A07	23	60	20.75		0.29
SS-UC23-A07	23	60	21.58	17.00	0.30
SS-UC23-A07	23	60	22.06	47.86	0.30
SS-UC23-A07	27	61	22.34	131.08	0.31
SS-UC27-A08	27	61	18.34		0.25
SS-UC27-A08	27	61	21.51	9.78	0.31
SS-UC27-A08	27	61	22.68	55.52	0.30
SS-UC78-A02	78	71	11.65		0.36
SS-UC78-A02	78	71	15.31	8.63	0.36
SS-UC78-A02	78	71	13.10	27.84	0.42
SS-UC78-A04	78	71	5.12		0.39
SS-UC78-A04	78	71	7.10	6.61	0.46
SS-UC78-A04	78	71	7.86	11.62	0.46
SS-UC127-A01	127	81	5.93		N/A
SS-UC127-A01	127	81	8.14	5.25	N/A
SS-UC127-A01	127	81	7.86	10.58	N/A
SS-UC127-A03	127	81	3.79		0.49
SS-UC127-A03	127	81	3.08	2.56	**N/A
SS-UC21-B02	21	71	11.65		0.38
SS-UC21-B02	21	71	14.62	25.99	0.37
SS-UC21-B02	21	71	13.86	40.69	0.38
SS-UC57-B03	57	80	7.58		0.41
SS-UC57-B03	57	80	9.17	4.12	0.39
SS-UC57-B03	57	80	8.76	9.11	0.44
SS-UC63-B04	63	81	5.86		0.46
SS-UC63-B04	63	81	7.72	6.72	0.45
SS-UC63-B04	63	81	7.17	11.34	0.44
SS-UC63-B05	63	81	4.36		0.27
SS-UC63-B05	63	81	6.23	2.34	0.33
SS-UC63-B05	63	81	8.27	10.85	0.44
SS-UC84-B06	84	86	5.25		0.44
SS-UC84-B06	84	86	6.86	4.35	**N/A
SS-UC84-B06	84	86	6.43	7.65	**N/A
SS-UC21-C01	21	81	13.03		0.33
SS-UC21-C01	21	81	14.55	21.24	0.34
SS-UC21-C04	21	81	N/A		N/A
SS-UC39-C02	39	86	1.38		N/A
SS-UC39-C02	39	86	6.37	1.65	N/A
SS-UC39-C03	39	86	2.21		0.16
SS-UC39-C03	39	86	10.89	2.36	0.40

N/A = Data not recorded during test

*Initial elastic properties do not list the axial stress where the modulus was calculated

**Poisson's ratio beyond sample dilation were excluded.