NBSIR 81-2422

Factors Affecting the Service Lives of Phase Change Storage Systems

December 1981

Issued February 1982

Prepared For:

U.S. Department of Energy
Office of Solar Heat Technologies
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U.S. DEPARTMENT OF COMMERCE, Malcolm Baldrige, ***Secretary***
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ABSTRACT

Phase change storage systems currently in use or in advanced stages of development are identified. Possible modes of degradation which may affect service lives are considered. Specifically, the effects of crystal growth, crystal segregation, supercooling, corrosion and thermal decomposition are discussed. The generic basis for the development of performance tests for inorganic phase change materials is described.

Key Words: crystal growth; encapsulants; failure mechanisms; nucleating agents; phase change storage; service life prediction.

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1. INTRODUCTION

An efficient method of storing solar energy is its conversion into the latent heat of fusion of a material which undergoes a reversible phase change in an appropriate temperature range. When such a material undergoes a phase change on heating, energy, as latent heat, is absorbed by the phase change material (PCM). Only after the phase change is complete and all the latent heat absorbed, will the temperature of the PCM again increase. When the PCM is allowed to cool, the reverse takes place with heat being released as sensible heat as the temperature drops and as latent heat during the phase change.

PCMs used in solar applications are typically either organic materials, such as paraffin wax, where the phase change is the melting and solidification of the wax, or inorganic salts, where the phase change usually involves dehydration. An example of the latter is sodium sulfate decahydrate ($Na_2SO_4 \cdot 10H_2O$) which is commonly called Glauber's salt. When Glauber's salt is heated from room temperature about 0.4 cal/gram-°C is absorbed. At 32°C a phase change occurs resulting in the formation of a saturated aqueous solution of sodium sulfate and a crystalline Na_2SO_4 precipitate. The latent heat absorbed in this phase change is about 60 cal/gram.

The advantages to using PCMs as energy storage media are twofold. First PCM storage systems are compact when compared with sensible heat storage units such as rock bins or water tanks. For example, about eight times more volume is required to store a given amount of heat using a water tank than is required for a sodium sulfate decahydrate PCM [12]. Similarly, the relative volume advantage over rock bed storage is about 17 to 1 [12]. A parallel advantage exists with respect to relative weight of the structured bins [12] and this allows much greater flexibility in choosing a location for the storage unit. The second advantage is that thermal stratification does not occur in phase change storage systems, because temperatures remain nearly constant throughout the charge-discharge cycle. If the melting point is chosen so that the storage unit provides heat at a temperature slightly above the minimum temperature required by the system, then the output from the solar collector need be only a few degrees warmer than the minimum temperature regardless of whether this storage unit is charged or discharged. By contrast, a sensible heat storage system typically operates at 22 to 33°C above its minimum operating temperature when it is fully charged.

2. IDENTIFICATION OF PCM'S AND ENCAPSULANTS

A review of the literature identified the PCM systems currently in use or with potential for widespread use and identified problems associated with the PCM's and their containment systems. A summary of the PCM systems currently in use or with future potential are given in tables 1 and 2. The status of development of the PCM storage systems, listed in these tables is divided into three groups: commercially available; advanced research/demonstration stage; and exploratory research stage. Based on this literature review the most promising systems appear to be paraffin wax encapsulated in steel or plastic, and salt hydrate encapsulated in plastic or in bulk form (tanks). Hydrated salts are not contained in steel due to corrosion problems. The types of PCM system are divided into two groups, organic and inorganic, and further divided into four types of encapsulation or containment: macroencapsulation in metal or plastic, microencapsulation, and bulk storage in tanks. Macroencapsulation is the containment of the paraffin wax or salt hydrate, such as $Na_2SO_4 \cdot 10H_2O$, in conveniently sized, easily handled containers. The containers may be long plastic tubes, "gallon" cans, plastic trays, foil pouches, sausage chubs or a variety of other packaging forms. Microencapsulation refers to packaging schemes in which small granules or pellets of PCM, about 1/8 inch in diameter, are packaged in individual envelopes. An example is the use of form-stable, high density polyethylene pellets approximately 1/8 inch in diameter as a PCM. The polyethylene is lightly crosslinked so that when it undergoes the phase change from a crystalline to an amorphous state the pellets retain their shape and form. The advantage of using small sized pellets is that the heat exchange between the PCM and the transfer fluid is improved. Bulk storage refers to the storage of PCM's in large tanks, equipped with heat exchangers.

Table 1

Typical Combinations of Encapsulating and Inorganic Phase Change Materials Presently Available or Being Considered for Thermal Storage [2, 3, 8-11]

Developers and Description of Systems

C = commercially available A = advanced demonstration stage R = research stage

Type of Encapsulant	System			
Plastic	Glauber's salt in rigid plastic containers. Problems with performance [C]	Ceiling tiles filled with Glauber's salt in plastic foil pouches [C]	Salt hydrate mixture in flexible plastic chubs [A]	Disodium phosphate dodecahydrate in rigid plastic. Water permeability a concern [R]
	$CaCl_2 \cdot 6H_2O$ eutectic mixture in high density polyethylene blackened tubes. [C]	Salt hydrates in rigid plastic trays. Problems with salt segregation [A]	$CaCl_2 \cdot 6H_2O$ in high density polyethylene tubes, 2-3 inch dia. [R]	Glauber's salt in rigid polyethylene trays [C]
Metal	Glauber's salt in stainless steel tubes [C]			
Bulk	Glauber's salt & immiscible heat transfer liquid in tank [C]	Salt hydrate in tank with heat exchanger [A]	Sodium acetate trihydrate with intermediate immiscible heat transfer fluid. No solid heat transfer surface [A]	Rotating drum with Glauber's salt inside with heat exchangers [R]
	Sodium thiosulfate pentahydrate in tank with plastic heat exchange tubes [C]	Various salt hydrates in tanks with high density polyethylene heat exchangers. Tested 1000 cycles [A]	Disodium phosphate dodecahydrate and missible heat transfer fluid, problems with surface [R]	
Micro-encapsulation	Glauber's salt crystals coated with 3% of encapsulating material, packaging in trays [C]			

Table 2. Typical Combinations of Encapsulating and Organic Phase Change Materials Presently Available or Being Considered for Thermal Storge [1-7]

C = commercially available A = advanced demonstration stage R = research stage

Type of Encapsulant	System		
Plastic	Poroplastic sheets impregnated with stearic acid-paraffin combination [R]		
Metal	Paraffin in 1 gal. steel cans. tested 3 yrs, few problems identified. [C]	Paraffin in beer cans [R]	Paraffin (M.P. 187 F°) stored in modular Al panels to supply process water. [C]
Bulk	None		
Macro-encapsulation	Form-stable crystalline polymer pellets (M.P. 120-140°C) in ethylene glycol. [R]	Form-stable, electron beam crosslinked polyethylene pellets. [R]	50-100 µm paraffin spheres encapsulated in polymeric materials. Used in a 40% solids-water slurry [C]

3. DEGRADATION OF PCM'S

3.1 DEGRADATION OF INORGANIC SALT HYDRATE PCM'S

3.1.1 Crystal Segregation - Life of Thickening Agents

Typical salt hydrate PCMs include $CaCl_2 \cdot 6H_2O$, $Na_2S_2O_3 \cdot 5H_2O$, $Na_2SO_4 \cdot 10H_2O$ and $Na_2HPO_4 \cdot 12H_2O$. $CaCl_2 \cdot 6H_2O$ and $Na_2S_2O_3 \cdot 5H_2O$ melt congruently. $Na_2HPO_4 \cdot 12H_2O$ melts semicongruently when the heptahydrate ($Na_2HPO_4 \cdot 7H_2O$) is present [2, 12] and congruently if it is not present. $Na_2SO_4 \cdot 10H_2O$ melts incongruently. A congruently melting salt is one which is completely soluble in its water of hydration at its transition temperature. When a salt is not completely soluble in its water of hydration, the undissolved salt can precipitate and settle to the bottom of its container where it cannot be easily rehydrated. If, however, the precipitated material becomes fully soluble in its water of hydration at a temperature a few degrees above its melting point, the material is said to melt semicongruently. If the precipitated material does not become fully soluble as the temperature is raised, the material is said to melt incongruently. Resistance to rehydration which results from incongruent melting is a major problem with salt hydrate PCM's. The problem is the most serious for $Na_2SO_4 \cdot 10H_2O$ and the least for $CaCl_2 \cdot 6H_2O$. This is because, while $CaCl_2 \cdot 6H_2O$ melts semicongruently in a technical sense, its behavior is very similar to that of a congruently melting salt [3].

The crystal segregation due to incongruent melting of the salt hydrates can be controlled by thickening agents. Thickening agents are additives used to fix the salt in a gel to eliminate settling. The effectiveness of the thickening agents listed in table 3 have been examined experimentally with a wide range of results [12, 13, 14]. The least expensive organic thickening agents, such as wood pulp, paper pulp, and starch [12, 13] appear to work well in short-term tests, but are believed to slowly decompose by bacterial or enzymatic action. The use of formaldehyde or other preservatives to prevent the decomposition has been suggested [14] but their effectiveness has not been verified experimentally. Other organic thickeners such as polyvinyl alcohol, hydroxethyl cellulose, and methyl cellulose also appear to work well in short duration tests but their cost is probably prohibitive [13]. Inorganic substances such as clays that can form thixotropic gels have also been used as thickening agents [12, 15]. When thixotropic materials are stirred or vibrated vigorously, they exhibit a relatively low viscosity which facilitates the initial container filling operation, but when left undisturbed a gel structure forms which hinders subsequent settling.

The thickening agents that appear to hold the most potential for commercial applications are inorganic thixotropic gels and stabilized wood pulp, starch and certain proprietary elastomeric materials. Test methodologies to evaluate the performance of thickening agents are needed.

3.1.2 Supercooling - Life of Nucleating Agents

Supercooling is prevalent in salt hydrate PCM's. As a salt hydrate PCM cools, its temperature can drop below its normal melting point without a change in

Table 3. Potential Thickening Agent

Organic	Inorganic
wood shavings	clays (montmorillonite type, attapulgite)
saw dust	diatomaceous earth
paper pulp	silica gel
methyl cellulose	
hydroxyethyl cellulose	
poly (vinyl alcohol)	
poly (acrylic acid)	
starch	
proprietary gels	

phase and without the release of latent heat. The temperature will continue to drop until crystals of solid PCM nucleate. At that point rapid crystal growth occurs accompanied by an increase in temperature. The problem associated with supercooling is that the system may cool below the point at which useful heat can be extracted. A method to nucleate PCM crystallization is usually required to prevent supercooling. The method commercially used is to add a nucleating agent to the encapsulated PCM to provide surfaces on which the salt hydrates will crystallize. Effective nucleating agents are crystalline materials whose interatomic lattice spacings are within 15 percent of that of the salt to be crystallized [16]. In addition, the nucleating agent must freeze at a temperature above the freezing point of the salt hydrate.

Nucleating agents have been found for the commonly used salt hydrates; however some of these agents are proprietary [12, 14]. A typical example of a salt hydrate/nucleating agent/thickening agent system is $Na_2SO_4 \cdot 0H_2O$ containing three to five weight percent $Na_2B_4O_7 \cdot 0H_2O$ (borax) as a nucleating agent [12] and silica gel as a thickening agent. At the freezing point of $Na_2SO_4 \cdot 10H_2O$ borax crystals are present and provide nuclei on which the $Na_2SO_4 \cdot 10H_2O$ crystals may grow. Because the density of borax is slightly greater than that of the saturated Na_2SO_4 solution, the borax nuclei will settle unless a thickening agent such as silica gel is present.

The life of a nucleating agent may be limited by a variety of factors. For example, when silica gel was used as a thickener, it was observed to react with borax and inhibit its nucleating capacity. Thus, both the settling of a nucleating agent and its reduction in nucleating capacity could limit the useful life of a nucleating agent and the thermal storage system. Test methods are necessary to evaluate the effectiveness of nucleation agents as well as the compatibility of the nucleating agent, thickening agent and phase change material.

3.1.3 Crystal Growth - Life of Salt Hydrates

The rate of growth of crystals of a hydrating salt will affect the rate of heat transfer and hence system efficiency. The crystal growth rate has been determined to be 0.055 cm/hr for $Na_2SO_4 \cdot 10H_2O$ for temperatures near the transition temperature [16]. Cyclic testing of $Na_2SO_4 \cdot 10H_2O$ using both wood pulp and attapulgite clay as thickeners has shown that large crystals of Na_2SO_4 have formed after about 200-300 cycles [13, 15]. Because the large crystals resist rehydration, a reduction in the system efficiency of as much as 40 percent results [15].

It is known that impurities can alter the crystal growth rate and the crystal habit [17]. In general, impurities decrease crystal growth rates. In light of this, the service life of salt hydrate, and more specifically, changes in crystal growth rate and growth habit with thermal cycling in thickened systems must be ascertained.

3.2 DEGRADATION OF ORGANIC PCM'S

Organic materials may be slightly less desirable as PCM's than inorganic salt hydrates due to lower heats of fusion, higher cost and the danger associated with the storage of large quantities of flammable substances. They do, however, melt congruently and generally do not supercool. This allows repeated thermal cycling with no apparent loss in thermal performance. The most promising organic systems appear to be paraffin waxes encapsulated in steel or plastic. The major problems with paraffin waxes are the relatively high degree of shrinkage upon solidifying and the low thermal conductivity of the solid waxes. Waxes undergo 5 to 15 percent shrinkage upon solidifying. When used in bulk, this shrinkage could result in void formation which would affect heat transfer within the system.

Little information regarding the long-term chemical stability of organic PCM's is available. Potential problems include the generation of volatile degradation products leading to bloating of the container, the generation of moisture and the generation of organic acids which would be aggressive towards the encapsulation materials. Test methods to assess these potential problems need to be developed.

4. DEGRADATION OF ENCAPSULATING MATERIALS

Metals with the exception of stainless steel have not, in general, been chosen as a containment material for commercially available systems or advanced research demonstration projects due to corrosion. Rather, salt hydrates are typically macroencapsulated in plastic materials and many bulk storage systems utilize plastic tanks and plumbing. Thermal storage systems using paraffin encapsulated in steel are not expected to develop corrosion problems. Paraffin, in general, is quite inert and corrosion may be a problem only if impurities or trace amounts of moisture are present. Waxes, however, are not compatible with polyethylene and polypropylene since they can cause stress cracking in these materials.

When plastics or plastic-foil laminated films are used to contain the a salt hydrate PCM, film puncture by salt hydrate crystals may be a problem [18]. The relatively slow rehydration of the salt, as would be expected during in-service cycling of the salt hydrate storage system, may produce preferential grain growth capable of penetrating the thin plastic film encapsulation. This phenomenon may not be evident from rapid cycling tests.

The potential problem of water migration through the plastic container walls has been cited in the literature [3, 12]. Water lost through the plastic container walls or container closures can not conveniently be replaced, and would in time reduce the system efficiency since insuffficent water would be available to completely rehydrate the PCM salts. However, values of moisture loss rates have been reported to be as low as 0.25 percent water per year [19] (through high density polyethylene). Other data on the rate of moisture loss through plastic is also available [20]. It is possible that rapid cyclic testing typical of much of the present solar energy research efforts may not reveal effects due to water loss or the settling of the PCM salts, both of which would occur in the time frame of real application [3].

5. DEVELOPMENT OF PERFORMANCE TESTS FOR PHASE CHANGE SYSTEMS

Based on the PCM and encapsulation materials currently in use, it is apparent that the technology is tending towards the utilization of salt hydrate PCM's. Typically, these systems are incongruently or semicongruently melting, inorganic salt hydrates in which the hydrated phase is heterogeneously nucleated on cooling below the transition temperature. Clay thickened, borax nucleated, Glaubers salt ($Na_2SO_4 \cdot 10H_2O$) encapsulated in plastic is one example of a system of this type. It is also apparent that there is a need for methods of test by which the durability of phase change systems of this type may be assessed. Current voluntary consensus standards methods may be applied to various physical property measurements. However, tests by which the long-term performance of phase change systems may be evaluated, both in terms of material degradation and in terms of loss of energy storage capacity, are less well developed.

Two major obstacles to the development of performance tests can be identified. First, the diversity of possible materials and system designs that must be taken into consideration require that test methods be sufficiently flexible to be relevant to unanticipated materials and system designs. Second, performance tests generally require both acceleration and simulation. Acceleration can usually be attained. However, simulation can be difficult if failure mechanisms are unknown or poorly understood. In spite of this, test parameters can be identified and constraints hypothesized based on the assumption that a performance test must involve thermal cycling. Figure 1 shows a simplified generalized cycle for a salt hydrate phase change system where:

T_{MAX} is the maximum temperature which occurs in the cycle,

T_H is the transition temperature on heating,

T_C is the transition temperature on cooling

T_{MIN} is the minimum temperature which occurs in the cycle.

α and α' are the rates at which the system is heated from T_{MIN} to T_H and from T_H to T_{MAX}, respectively.

β and β' are the corresponding cooling rates.
t_H and t_C are the times spent at T_H and T_C, respectively.
t_{MAX} and t_{MIN} are the times spent at T_{MAX} and T_{MIN}.

It is possible to qualitatively consider the response of a phase change material in terms of the above parameters based on the cycle shown in figure 1. T_{MAX} must be above the transition temperature on heating, T_H, and T_{MIN} must be below the transition temperature on cooling, T_C. The latter may be the case initially, but may not obtain after N cycles. One failure mechanism involves the depression of T_C due to the persistence of crystals of anhydrous material at increasingly lower temperatures resulting from preferential crystal growth or loss in effectiveness of nucleating agents.

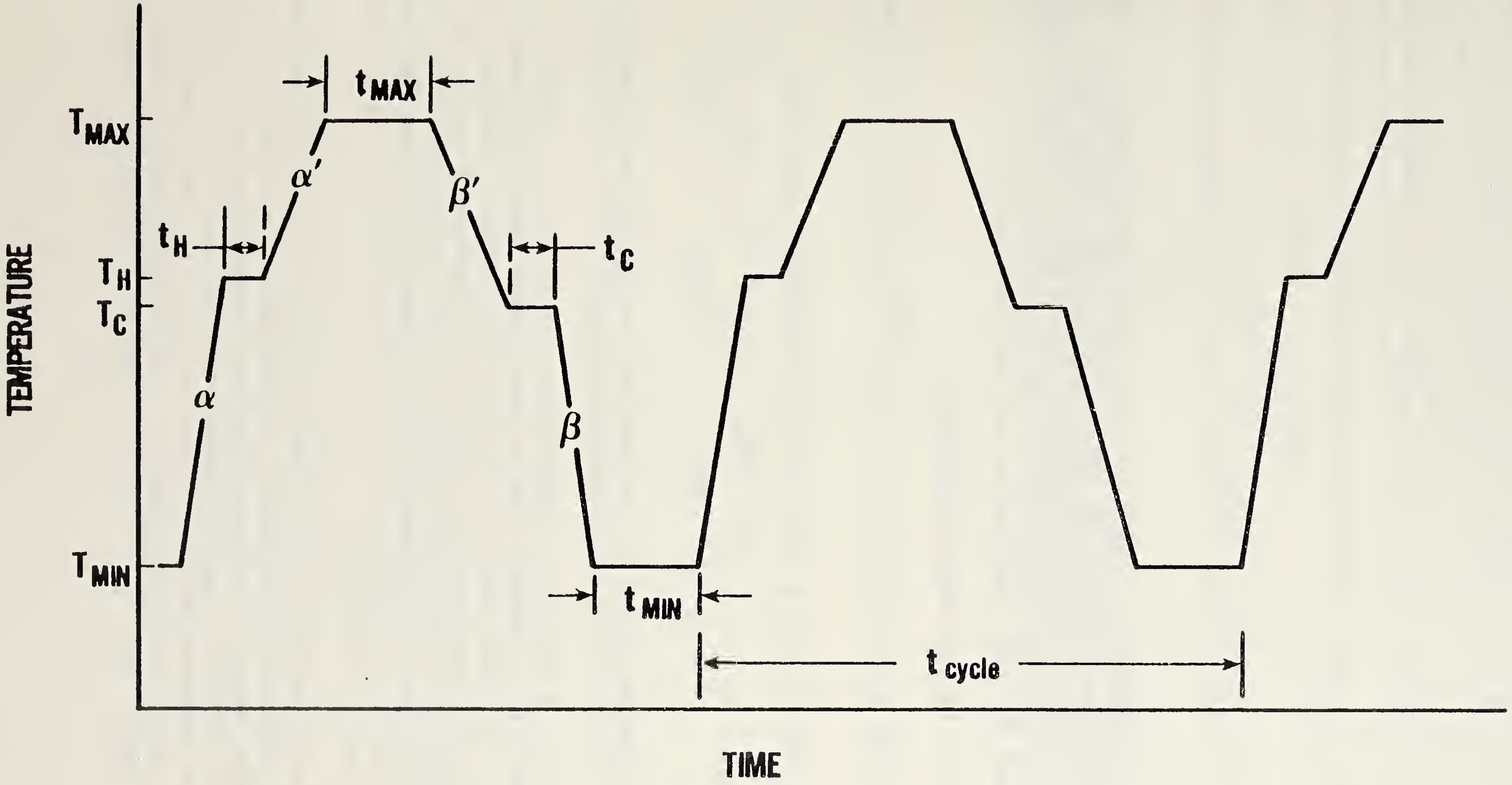

Figure 1. A generalized time-temperature cycle for a salt hydrate phase change system

Therefore, the variation in T_C, with the number of cycles provides a diagnostic tool for assessing one mode of failure. In addition, time spent above T_H must be greater than the time required for complete melting, i.e.,

$$t_H + t_{MAX} + \left(\frac{T_{MAX} - T_H}{\alpha'}\right) - \left(\frac{T_{MAX} - T_C}{\beta'}\right) > t_{\text{complete melting}}, \text{ and}$$

similarly, the time spent below T_C must be greater than the time required for complete freezing, i.e.,

$$t_C + t_{MIN} + \left(\frac{T_H - T_{MIN}}{\alpha}\right) - \left(\frac{T_C - T_{MIN}}{\beta}\right) > t_{\text{complete freezing}},$$

if the maximum amount of heat is to be stored and delivered. These criteria have material dependencies in that t_H and t_C may increase with an increasing number of cycles. This is because t_C and t_H depend on crystal size distributions which will be affected by the heating and cooling rates, and by the maximum and minimum temperatures and their durations.

The total cycle time may be defined as

$$t_{cycle} = t_{MAX} + t_{MIN} + t_H + t_C + \left(\frac{T_H - T_{MIN}}{\alpha}\right) + \left(\frac{T_{MAX} - T_H}{\alpha}\right) - \left(\frac{T_{MAX} - T_C}{\beta}\right) - \left(\frac{T_C - T}{\beta}\right.$$

This, in turn, allows a definition of a minimum cycle time:

$$t_{cycle,\ min} = t_H - t_C + \left(\frac{T_H - T_{MIN}}{\alpha_{MAX}}\right) + \left(\frac{T_{MAX} - T_H}{\alpha'_{MAX}}\right) - \left(\frac{T_{MAX} - T_C}{\beta'_{MAX}}\right) \left(\frac{T_C - T_{MIN}}{\beta_{MAX}}\right)$$

where the maximum values of the heating and cooling rates are determined by the thermal conductivities of the system components and the system geometry.

The response of the phase change material will vary according to the shape of the thermal cycle imposed. The average crystal size will show a dependence on α' and β. If these rates are low the system spends relatively more time near the transition temperatures. Under this circumstance crystal nucleation is less favorable but the growth rates of those crystals which do form will be relatively higher. This results in the formation of fewer but larger cyrstals. Alternatively, if heating and cooling rates are high, a larger number of smaller crystals will form.

The maximum temperature will affect the size distribution and population of anhydrous crystals in two ways. The maximum cycle temperature will determine the relative amount of anhydrous material present through solubility considerations. In addition, T_{max} in combination with the total time spent above the transition temperatures will affect the size distribution of both the anhydrous material and the nucleating agent. Coarsening of larger crystals and dissolution of smaller ones will occur with time at a temperature dependent rate.

This phenomenon, called Ostwald ripening, occurs because only particles of one size can be in equilibrium with the solution at a given concentration. Over time this may result in a decrease in the total number of nucleation sites on which the hydrated salt may crystallize. Further, because this phenomenon also results in an increase in the average size of the anhydrous crystals, the time required for complete transformation to the hydrated salt will increase. If this time exceeds

$$[t_{MIN} + t_C + (\frac{T_H - T_{MIN}}{\alpha}) - (\frac{T_C - T_{MIN}}{\beta})] \quad ,$$

the fraction of energy stored which is delivered to the environment will decrease.

Because optimal test methods should meet the criteria of acceleration and simulation, it becomes possible to identify and model failure mechanisms. Once this is accomplished and the relationships between the system performance and the test parameters have been developed, service life prediction may be achieved.

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NBS-114A (REV. 2-8C)

U.S. DEPT. OF COMM.

BIBLIOGRAPHIC DATA SHEET *(See instructions)*

1. PUBLICATION OR REPORT NO. NBSIR 81-2422

2. Performing Organ. Report No.

3. Publication Date February 1982

4. TITLE AND SUBTITLE

Factors Affecting the Service Lives of Phase Change Storage Systems

5. AUTHOR(S)

John B. Ings and Paul Wencil Brown

6. PERFORMING ORGANIZATION *(If joint or other than NBS, see instructions)*

NATIONAL BUREAU OF STANDARDS
DEPARTMENT OF COMMERCE
WASHINGTON, D.C. 20234

7. Contract/Grant No.

8. Type of Report & Period Covered

9. SPONSORING ORGANIZATION NAME AND COMPLETE ADDRESS *(Street, City, State, ZIP)*

U.S. Department of Energy
Office of Solar Applications for Buildings
Conservation and Renewable Resources
Washington, DC 20585

10. SUPPLEMENTARY NOTES

☐ Document describes a computer program; SF-185, FIPS Software Summary, is attached.

11. ABSTRACT *(A 200-word or less factual summary of most significant information. If document includes a significant bibliography or literature survey, mention it here)*

Phase change storage systems currently in use or which are in the advanced stages of development are identified. Various possible modes of degradation which may affect service lives are considered. Specifically, the effects of crystal growth, crystal segregation, supercooling, corrosion and thermal decomposition are discussed. The generic basis for the development of performance tests for inorganic phase change materials is described.

12. KEY WORDS *(Six to twelve entries; alphabetical order; capitalize only proper names; and separate key words by semicolons)*

Crystal growth; encapsulants; failure mechanisms; nucleating agent; phase change storage; service life prediction.

13. AVAILABILITY

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14. NO. OF PRINTED PAGES 19

15. Price $6.00

USCOMM-DC 6043-P80