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Thermal Energy Storage Technical Progress Report April 1986–March 1987

J. J. Tomlinson
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THERMAL ENERGY STORAGE TECHNICAL PROGRESS REPORT
APRIL 1986—MARCH 1987

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ABSTRACT

Progress in the development of Thermal Energy Storage (TES) technology under the Oak Ridge National Laboratory TES Program for the period April 1986--March 1987 is reported. Program goals and project structure are presented. Each of the projects active during this report period is discussed, and the interrelation of specific projects toward major technology transfer activities is described.

1. PROGRAM OVERVIEW

This report presents the status of projects active under the Oak Ridge National Laboratory (ORNL) Thermal Energy Storage (TES) Program during the 1-year period ending March 31, 1987. ORNL is the principal research laboratory for the development of diurnal and industrial TES technologies, acting as field manager for the Department of Energy (DOE), Office of Energy Storage and Distribution. The application areas of the program are those of industrial reject heat utilization, and energy conservation through demand-side electric utility load leveling, and passive and active solar energy storage. The ORNL program is conducted in consultation with, and with the concurrence of, DOE.

The purpose of the TES program is to perform research and development (R&D) in critical, high-risk technology areas and to transfer the results to the private sector so that advanced, high-performance TES systems may be developed and successfully marketed for specific applications. This purpose is expressed by the overall goals of the TES program: to (1) create the technology base required for development of efficient, cost-effective TES concepts in diurnal heating and cooling and industrial applications and (2) work with industry, universities,

technical societies, and trade associations to transfer the technologies to the private sector.

In pursuit of this goal, the following five program objectives have been identified as representing the critical areas for resource allocation. Each objective is associated with a benefit to the technology of TES, and the necessary targets with which to measure progress toward achieving the objectives have been established. The strategy followed is to initiate projects that address technical problems that must be overcome to meet these targets.

1. Develop TES concepts and media that provide both heat (winter) and cool (summer) storage for heat pump systems.

Benefit: Improve heat pump applicability for utility load control through performance that equals or exceeds nonstorage heat pump performance.

Targets: economic — \$22/kWh (\$6700/10⁶ Btu) installed cost (1986 dollars).

technical — Heat exchange effectiveness of 0.95; storage temperature: 7 to 12°C (45 to 53°F) cool; 38 to 49°C (100 to 120°F) hot; round-trip efficiency of 90%.

2. Develop TES concepts for capture, storage, and reuse of high-temperature energy that significantly increase the utilization of industrial reject heat.

Benefit: Make economically feasible the conservation of the significant energy available from industrial waste heat.

Targets: economic — \$28/kWh (\$8300/10⁶ Btu) installed cost (1986 dollars)

technical — Storage temperatures of 175 to 1100°C (350 to 2000°F); round-trip efficiency of 85%.

3. Develop TES concepts for industrial chill storage

Benefit: Improve electric utility capacity factor and reduce utility consumption of oil and gas through load shifting.

Target: economic — \$48/kWh (\$14,000/10⁶ Btu) installed cost (1986 dollars).

technical — Storage temperatures range: -18 to -46°C (0 to -50°F).

4. Perform research to advance the understanding of new TES materials and processes.

Benefit: Identify advanced concepts with improved technical and economic characteristics.

Targets: economic — Assess projected system costs.

technical — Determine physical and thermal properties and conceptual system descriptions.

5. Interact with commercial/industrial sector directly (workshops) or through technical societies and trade associations to evaluate research needs and to effect transfer of the technologies developed.

Benefit: Fulfill TES program goals through transfer of developed technologies to industry for implementation.

Targets: Wider recognition of the value of thermal storage by the private sector.

That the usefulness of TES is becoming more widely recognized is apparent from the recent introduction of commercial cool storage systems into large buildings for electrical load management. Data from these installations indicate that the performance (efficiency) of these systems is lacking. The results indicate that the systems are economic only with utility subsidies, and they use appreciably more energy than conventional cooling systems. Since electric utility regulatory bodies are quite sensitive to energy penalties associated with implementation of new technologies, the benefits of commercial cool storage could be short-lived unless efficiencies are increased. Thus, there is need for basic R&D on TES media and systems with the goal of enabling TES systems to perform the required function efficiently.

TES media that undergo a change of state (e.g., phase, structure, or chemical composition) at a desired storage temperature are required for improvement of present storage systems. Additionally, the economics of these storage media must be considered because the ultimate goal of

the TES program is successful technology transfer. Further, the elements that make up the reliability of a workable system must be addressed. The results of previous years' R&D in this program are seen in the identification of these materials that have good prospects for meeting the requirements of a viable TES system:

1. clathrates, double clathrates, semiclathrates, and related compounds that show two transition temperatures, thereby providing cool storage for air conditioning and heat storage for heating;
2. salt hydrates and their containment materials that allow integration into a passive solar structure, thereby allowing lightweight, practical building components to replace massive passive solar walls;
3. multicomponent, high-temperature phase-change materials (PCM) that allow direct contact heat exchange for industrial applications; and
4. new technologies that exploit slurries for increased heat transport in piping systems.

Within this context, the ORNL TES Program is focused on the identification and solution of the problems of TES for both building heating and cooling and industrial applications. Specific program activities are designed to extend the technology for TES systems by

1. developing advanced high-temperature storage media for industrial applications,
2. investigating complexing TES systems suitable for heat pump applications,
3. conducting laboratory materials testing to identify appropriate TES media materials,
4. performing technical and economic evaluations to identify TES developmental requirements and R&D objectives, and
5. evaluating new concepts for TES/heat transport.

The total scope of the program is shown in Fig. 1, which is the work breakdown structure (WBS) of the ORNL program. From this comprehensive structure, and by a process of prioritization, including budget constraints, and a consideration of the capabilities of the research community, certain projects were funded. The funded activities are

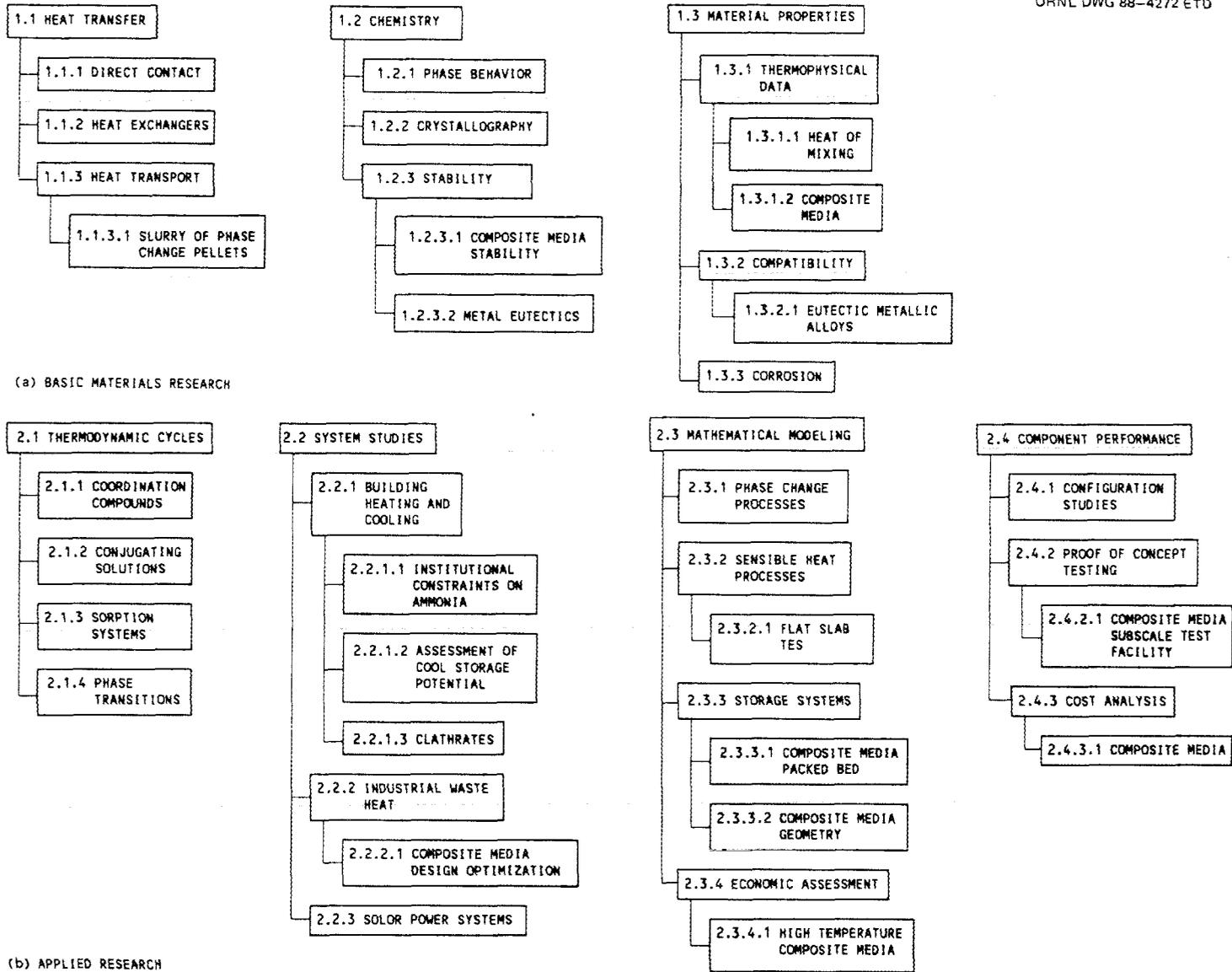


Fig. 1. Work breakdown structure of ORNL/TES program.

illustrated as shaded areas in Fig. 1 and are presented in the following sections of the report.

1.1 Basic Materials Research

1.1.1 Heat transfer

Adequate heat transfer, a fundamental requirement in any TES system, is of particular concern in systems in which thermal storage is effected through freezing and melting. In this case, heat transfer through the solid material is conduction-limited and is controlled by the TES medium surface area exposed to the working fluid. Insufficient surface areas either limit the heat transfer, or meet the heat transfer requirement at the expense of large temperature gradients between the TES material and the working fluid. Based on the potential of direct contact and related techniques for heat transfer enhancement, a program to develop an advanced cool storage (ice) system was initiated as shown in WBS project 1.1.1. The objective of this program is to develop a production and storage system in which ice (or ice-like material) is formed in the bulk of the water rather than on the solid heat transfer surface. Such a system may be based on the use of an intermediate heat transfer fluid, direct contact between a refrigerant and water, vacuum freezing, homologous seed crystals, control of the thermal-hydraulic boundary layer, or other innovative schemes. From a range of proposed concepts one will be chosen to be carried from laboratory studies through prototype design and construction and field evaluation.

Work package 1.1.3.1 consists of research in the enhanced heat-carrying capability of fluids slurried with solids that can undergo solid-solid phase change. The objective of the project is to establish proof-of-principle for improved TES performance by utilizing combined mechanisms of enhanced heat transfer, heat transport, and TES associated with a phase-change slurry used as the system working fluid. Preliminary concept and background development work already completed and reported shows that using a phase-change slurry as the thermal system working fluid (i.e., to remove thermal energy from the heat source and to transport it to, and reject it at, the heat sink) can potentially

yield considerable reduction in system heat transfer surface area and/or pumping power while increasing TES efficiency and end-use temperature.

Experiments to determine heat transfer enhancements due to a slurry flowing in a pipe were initiated. Preliminary results indicate that the slurry causes a significant increase in Nusselt number over pure water. Use of friction-reducing additives were found to greatly reduce slurry pumping power requirements.

1.1.2 Chemistry

An advanced material that has been under development by the TES Program is a composite consisting of salt eutectica contained in the interstices of a nonmelting ceramic. The composite material formed allows the latent heat of the eutectica to be utilized while the system retains its shape as a solid. Pellets of this material are suitable for packed-bed storage systems with the resulting efficiency of direct contact heat exchange with the working fluid. For high temperature applications, a sodium/barium carbonate eutectic contained in a magnesium oxide matrix has been studied.

The purpose of WBS project 1.2.3.1 is to quantify the metallurgical stability of the high temperature composite storage material. Previous small scale experiments have identified a tendency for the sodium-barium salt to leave the magnesium oxide support matrix and migrate the length of the packed bed. This migration phenomena results in decreased thermal performance and its long-term behavior is of some concern. During the previous year, the plan for studying this behavior shifted from laboratory scaled experiments to a sub-scale test facility which will be installed in a brick plant during FY-88.

The objective of WBS project 1.2.3.2 is to develop a TES medium consisting of an integral outer shell with a high melting temperature and an internal core of latent heat storage material with a lower melting temperature. The outer shell is to be inert and act as an encapsulation of, and corrosion protection from, the latent heat storage material.

Metallic eutectic alloys have long been of interest for use as TES media. Their high thermal conductivity (on the order of 200 J/m-s-°C)

and heat of fusion (300 to 500 J/g) make them excellent candidates. However, commercial use of alloy systems requires expensive containment in corrosion-resistant tubing. A solution to this problem is to develop a means of achieving an impermeable coating of the metallic eutectics so that a packed bed of spherical shot of the storage medium can be used in direct contact with the heat transfer fluid. Using silicon-based alloys, it is possible to achieve a heat storage shot with a relatively high-temperature, corrosion-resistant outer shell and a lower-temperature eutectic alloy inner core.

1.1.3 Material properties

The objective of WBS project 1.3.1.1 is to identify and explore the use of partially miscible liquids to store thermal energy through differences in the heats of mixing of the solution above and below its critical solution temperature. Several studies were conducted to identify possible binary liquid systems as TES candidates. For combined heat and cool storage applications, mixtures of water and an organic component with an upper critical solution temperature of $\sim 50^{\circ}\text{C}$ were recommended. For cool storage, the system would form a solution of ice and the liquid component at about 0°C . For cool storage alone, an aqueous system with a critical solution temperature of $\sim 5^{\circ}\text{C}$ can also be used for temperatures in the range of $2\text{--}10^{\circ}\text{C}$.

In the first phase of this investigation, analytical procedures were developed to estimate heats of mixing of liquid-liquid systems. The temperature variation of the equilibrium phase compositions is utilized in conjunction with activity coefficient models for liquid mixtures. These procedures were then used to identify mixtures with suitable properties for thermal energy storage applications. In order to confirm these results, heats of mixing will be measured experimentally for a number of potential systems in the second phase of this investigation.

Development of a medium temperature composite thermal energy storage material was begun under WBS project 1.3.1.2. The purpose of this effort was to define a set of materials that would store latent heat

energy in the 260—510°C (500 to 950°F) temperature range. The justification for this work is that a packed bed TES unit with only one PCM (i.e., one melt temperature) has little economic advantage over a traditional sensible heat storage system. This trend has been recognized for some time, and the development work on the high temperature $\text{Na}_2\text{CO}_3\text{-BaCO}_3/\text{MgO}$ composite had matured to the point where it was appropriate to begin developing a family of composite materials. During the previous year, two material combinations were identified, fabricated, and partially characterized. Further development was postponed pending the completion of design optimization studies. These studies were needed to confirm the initial operating range of 260 to 510°C, or to indicate a more appropriate temperature range.

The experimental determination of the rate of diffusion of silicon into an aluminum-silicon alloy is the subject of in-house research conducted under WBS project 1.3.2.1. The objectives of this research are to determine the silicon shell thickness required to retain a liquid aluminum-silicon alloy and to determine shell stability in the presence of phase redistributions occurring during melting and recrystallization.

Silicon-encapsulated shot of aluminum-silicon or other similar eutectics offers an extremely attractive possibility for direct-contact, high-temperature, latent heat storage. Successful development of pellets of one or more of these materials would make available storage media with a high latent heat of fusion, high heat conductivity, and corrosion resistance. However, the question of stability of the silicon shell to redistribution during repeated melting and recrystallization must be addressed to ensure that a practical material will result from such development efforts. This project provided experimental data on the nature of the material interface and determined conditions for media stability.

1.2 Applied Research

1.2.1 Thermodynamic cycles

Work package 2.1.1 concerns the study of generic problems of solid/vapor TES systems. Of interest are chemical systems that are

based on adsorption/desorption of ammonia with solid salts. Prior work to develop sorption TES systems has shown that, since diffusion of ammonia vapor into the salt during sorption is a surface phenomenon, large (and expensive) heat exchangers are necessary for suitable heat/mass diffusion rates. In the reporting period, research was initiated to evaluate inert solvents which function as holding media for a finely-divided adsorbent. As the solvent greatly increases the adsorbent surface area, heat exchanger requirements are reduced to the point that successful development of a working TES system based on solid-vapor sorption principles may be possible. Results of these investigations are to be released pending resolution of patent issues.

1.2.2 System studies

Ammonia is an excellent refrigerant and cool storage systems based on ammonia have the potential for high storage densities and operating flexibilities over a wide range of temperatures. However, corrosivity and toxicity issues associated with ammonia use have led to institutional regulations that may limit its use in certain applications. Work package 2.2.1.1 is an assessment of the regulations that have a bearing on ammoniated cycles. The results of this assessment are used to guide ammoniated TES system development.

Clathrate research has foundered on economics and on growing environmental concerns associated with chlorofluorocarbon refrigerants that have been studied for use in clathrate systems. Work package 2.2.1.3 is an evaluation of the potential returns from further clathrate research in light of these economic and environmental issues.

A comprehensive plan for the development, testing, and evaluation of phase change energy storage materials for capture and use of industrial waste heat was defined and partially implemented. This redefinition and redirection was necessitated by several factors which combined to significantly increase the scope and complexity of the Industrial TES Project. These factors included: (a) the designation of the high temperature 710°C composite storage media as the material to be used in proof of principle field tests, (b) the work to develop a medium temperature composite storage media, (c) the addition of Battelle

Pacific Northwest Laboratory contributions to the project management team, and (d) the decision to use economically based, design optimization studies as the primary decision making tool to determine the technical and economic feasibility of latent heat storage media. WBS project 2.2.2.1 represents the design optimization studies which are an integral part of the redirected Industrial TES Project. The technical and economic information needed to conduct these studies as well as the responsibilities for generating them were completely defined and partially implemented.

1.2.3 Mathematical modeling

Work package 2.3.3.1 is modeling task required for the high temperature TES design optimization studies. The ultimate result of these studies is a comparison between an optimum latent heat storage system (using the most current latent heat media and/or media geometry) and an optimum sensible heat storage system. The flat slab model produced by this task will be used to define the comparison sensible heat TES system.

As previously mentioned, the redirected Industrial TES Project will utilize design optimization studies as the primary decision making tool regarding the technical and economic feasibility of latent heat energy storage materials. Work packages 2.3.3.1 and 2.3.3.2 represent individual mathematical models required to conduct these studies. Project 2.3.3.1 is a task to define and build a packed bed model which utilizes cylindrically shaped pellets of the IGT composite media. The model will be used for (a) the generic optimization studies to be conducted by ORNL, (b) a separate design optimization study to define a full-scale TES system for a field test planned for late FY-89, and (c) to reduce data from a sub-scale test facility planned for the summer of 1988. Specific model performance requirements have been defined in the form of a work statement and work is currently underway.

Work package 2.3.3.2 is a study to define an optimum configuration for the composite media. Alternate geometries to the sphere and cylinder, as well as a range of different characteristic lengths for the spherical and cylindrical geometries, will be defined and characterized

in an effort to determine the one configuration that is the most efficient at storing latent heat energy. Personnel to conduct the study have been identified and a preliminary problem statement has been defined.

A preliminary economic feasibility assessment of latent heat storage systems using the composite was conducted under the WBS project 2.3.4.1. These assessments, based on present worth economic calculations for TES systems, focused on applications within the brick and tile industry. Results indicate that the composite media is economically superior to sensible heat media for moderately short cycle times. As a part of the redirected Industrial TES Project, future economic assessments will be incorporated into WBS project 2.2.2.1.

Work package 2.4.2.1 represents those activities associated with the sub-scale test facility. This field test is a major component of the redirected Industrial TES Project and contains many elements from the Basic Materials Research and Applied Research activities shown in Fig. 1. Each of the activities required to define and conduct the field test have been defined and are currently being implemented.

One of the most critical pieces of information required to conduct TES design optimization studies is the unit cost of the composite storage media. This cost is also the most uncertain variable in that the media is not currently being produced. Work package 2.4.3.1 represents a probabilistic cost analysis to determine the most likely future cost of the composite media. Efforts are currently underway to procure funding and identify potential subcontractors to conduct the study.

2. TECHNICAL PROGRAMS

There was a good balance between basic research in TES materials and applications studies this report year. All seven categories representative of the program included by active projects. Several projects are poised for major technology transfer activities in the near future.

2.1 Slurry Heat Transfer (WBS 1.1.3.1)

The primary objective of the first phase of the Slurry Heat Transfer Study is to establish proof-of-concept of enhanced slurry heat transfer relative to heat transfer in a pure single-phase liquid. Scoping studies have predicted that, under some conditions, the addition of solid particles to a liquid carrier enhances the heat transfer characteristics of the working fluid. An even greater improvement in heat transfer is postulated when the particles undergo a phase change during the heat transfer process. The use of phase change slurries as energy transmission fluids in a variety of thermal systems, also holds the promise of enhancing system performance, as well as reducing the size of associated hardware such as piping, pumps, heat exchangers, and storage tanks. The degree of heat transfer enhancement is a function of the properties of both the phase change material and the carrier fluid. Particle size, density, and particle loading are some of the important parameters that affect slurry hydrodynamics and heat transfer.

The initial proof-of-concept experiments have been designed to use pellets of cross-linked high density polyethylene (X-HDPE) slurried in a water glycol mixture. These particles undergo a phase transition at $\sim 132^{\circ}\text{C}$ (270°F) with a corresponding latent heat of 73 Btu/lb (169 kJ/kg). Since the molecular structure of the particles has been cross-linked using electron beam irradiation, they remain form-stable (i.e., retain their shape) during the phase transition.

An experimental facility to test the concept was designed and constructed at Argonne National Laboratory. The pumped facility consists

of supply and discharge tanks connected by a heated test section (stainless-steel pipe) through which the slurry is pumped. Instrumentation consists of 12 thermocouples and 6 voltage taps spaced along the pipe and pressure taps located at the test section entrance and exit to determine overall pressure drop.

The slurry heat transfer test program consists of three phases: (1) heat transfer tests with pure water, 0% particle loading; (2) heat transfer tests with a slurry of water and X-HDPE without phase change; and (3) heat transfer tests with a X-HDPE slurry phase change. Experiments have been conducted in the first two phases of the test program.

A matrix of nominal test conditions for the tests for pure water is presented in Table 1. The flow rates, velocities and corresponding pressure drops are typical values corresponding to the various speed settings on the pump (setting number form 1 to 8). The calculations of flow velocities and Reynolds number are based on the water properties at 38°C (60°F). The power requirements, electric currents and voltage drops are estimated by assuming bulk fluid temperature rises of 60°C (10°F) and 20°F (11.1°C) in the test section. The heat inputs required for a bulk fluid temperature rise of 10–20°F in the test section resulted in nominal inner wall-to-bulk temperature drop of 10–20°F within the desired range of flow rates (Table 1). The bulk fluid temperature rise in every subsection was calculated very accurately based on an energy balance. Analysis of data from slurry tests with HDPE was complicated by property differences between X-HDPE and water, and the fact that the two phases may not be in thermal equilibrium. The specific heat of X-HDPE is approximately half that of water. A simple one dimensional thermal conduction calculations revealed that 1.6 s is required to heat the centers of 1/8 in. diam X-HDPE particles to one half the surrounding fluid temperature and 3.5 s is needed to bring the center temperature up to 90% of the fluid temperature. Hence, during slurry heating, the temperature of the particles lagged behind that of the fluid. Based on the average tube flow velocity and test section length, the slurry was not in thermal equilibrium unless the test section flow was less than 100 lb/min. The current study produced three models for predicting the heat transfer coefficients of slurry flows.

Table 1. Test matrix for slurry heat transfer experiments without phase change^a

Pump setting No.	Flow rate (GPM)	Flow rate (lb/min)	Velocity (ft/s)	Pressure drop (psi)	Re	Power (kW) ΔT = 10-20°F	Current (A) ΔT = 10-20°F	Voltage drop (V) ΔT = 10-20°F
1	5.6	46.8	2.59	0.27	2.75×10^4	8.22-16.44	315-446	26.1-36.9
2	7.9	65.4	3.62	0.50	3.85×10^4	11.50-23.01	373-527	30.8-43.6
3	10.2	84.8	4.69	0.79	4.99×10^4	14.90-29.80	424-600	35.1-49.7
4	13.3	110.2	6.09	1.26	6.48×10^4	19.36-38.71	484-684	40.0-56.6
5	16.9	140.2	7.75	1.95	8.25×10^4	24.64-49.28	545-771	45.2-63.9
6	21.2	175.7	9.72	2.94	1.03×10^5	30.76-61.52	609-862	50.5-71.4
7	25.9	214.4	11.86	4.20	1.26×10^5	37.63-75.27	674-953	55.8-78.9
8	34.8	288.2	15.94	7.15	1.70×10^5	50.77-101.54	783-1107	64.8-91.7

^aBased on water properties at 100°F.

Two models (No Particle Heating and Thermal Equilibrium) are limiting cases and served to set the high and low bound on the heat transfer coefficient calculation. The third model is a Partial Thermal Equilibrium Model which estimates the degree of thermal equilibration between particle and water for the slurry system.

Information from the pure water tests were used to check both No Particle Heating and Thermal Equilibrium Model and to develop a basis for the Partial Thermal Equilibrium Model. As expected, the Partial Thermal Equilibrium Model provided the best match with measured temperatures in the single phase tests.

The Nusselt number for slurry heat transfer when plotted versus a modified slurry Reynolds number based on an effective viscosity, was found to be larger than that for pure water and to increase with an increasing particle volumetric loading. Significant Nusselt number enhancement can be observed from Fig. 2. Within the entire Reynolds number range with the 35% loading, the Nusselt number is increased by a factor of 2.5 or more.

As shown in Fig. 3, the measured Stanton number consistently showed a greater value than that of pure water at all ranges of flow rate. At loading 30% the heat transfer enhancement observed is 6% at 70 lbm/min to 12% at 190 lbm/min.

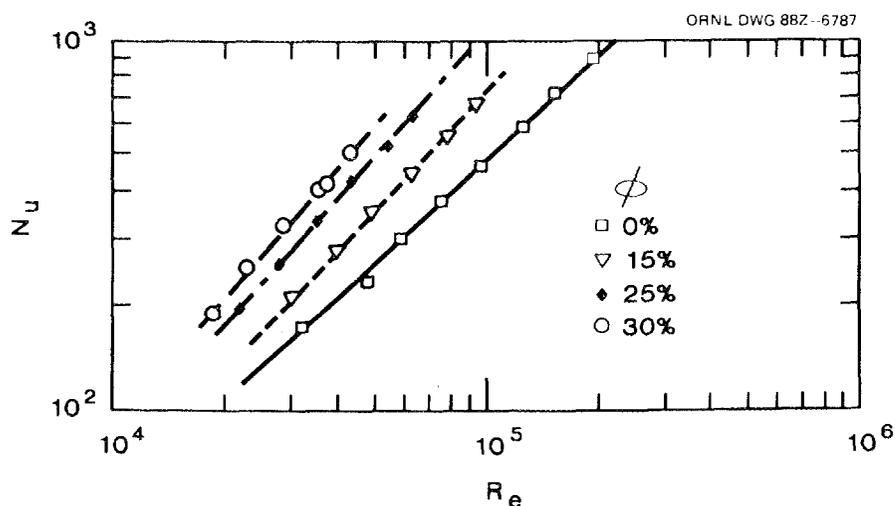


Fig. 2. Heat transfer correlation for X-HDPE/water slurry over range of particle loadings, ϕ .

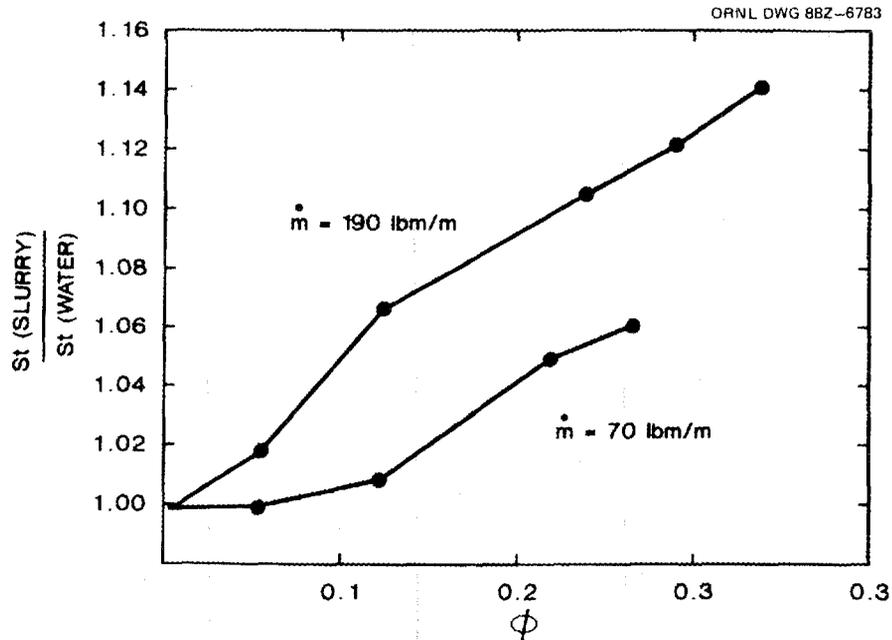


Fig. 3. Slurry heat transfer enhancement as determined by Stanton number, St and particle loading, ϕ .

Further work is underway to resolve the differences in slurry heat transfer as characterized by Nusselt and Stanton numbers.

2.2 Stability of Encapsulated Metal Eutectics (WBS 1.2.3.2)

Mobley and Rapp¹ have pointed out the potential advantages of metallic alloys as phase change heat storage materials for recovery of industrial waste heat. In particular, they proposed the development of methods to encapsulate aluminum-silicon alloy shot in an integral silicon shell intended to contain the heat storage alloy during repeated cycles of melting and freezing of the eutectic core. Their initial proposal was to form silicon encapsulated shot by cooling a molten aluminum-silicon mixture of hypereutectic, or silicon rich, composition.

The concept may be illustrated with reference to the aluminum-silicon equilibrium phase diagram shown in Fig. 4. The silicon encapsulated shot would have an overall composition (core and shell) as indicated by the line A-B in the phase diagram, somewhat richer in silicon than the eutectic composition. If heated to temperatures above that of

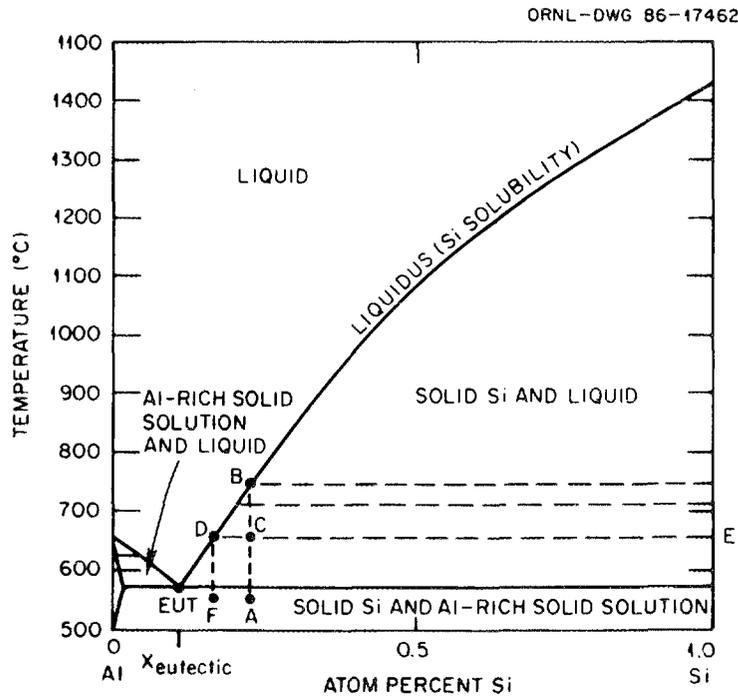


Fig. 4. Al-Si equilibrium phase diagram.

point B, a homogeneous liquid solution would result. On cooling the melt, silicon would precipitate as the temperature fell between T_B and T_D while the equilibrium composition of the melt within the solid silicon shell changed from X_B to X_D along the liquidus line. As the temperature continued to fall, between T_D and T_F , additional silicon would precipitate; the composition of liquid would be further depleted in silicon until the composition X_{eut} was reached at temperature T_{eut} . Here the melt is saturated with both Si and Al. On further heat removal the eutectic of Si and of Al-rich solid solution will precipitate in the fixed, eutectic ratio, with no change in composition (or temperature) of the remaining liquid until no more liquid remains. The latent heat evolved in the solidification is the basis of the energy storage. The temperature of the solid phase will then fall as further heat is removed.

If the shot is reheated to T_B , or above, it will liquefy. If however, as in the intended application, the shot is heated only to some lower temperature, T_C , some solid silicon will remain, which could form

an integral shell surrounding the core of eutectic composition. As the shot is heated from T_A to T_{eut} no melting occurs. At T_{eut} the eutectic core melts, the relative amounts of solid silicon and of liquid eutectic being in the ratio $(X_A - X_{eut})/(1-X_A)$. On further heating, the composition will change along the liquidus curve. At T_C the ratio of solid to liquid will have decreased to $(X_A - X_D)/(1-X_A)$, but a solid silicon shell could conceivably remain, under proper conditions, if the temperature remained below T_B . The phase diagram, however, indicates only the relative amounts of the two phases at equilibrium. It does not indicate their spatial distribution. Therefore, if a mechanism exists for the precipitating silicon to deposit preferentially on one side of the shot, e.g., because of temperature gradients or gravity, the remainder of the shot would be poorer in silicon. If the local composition of part of the shot changed to X_D , for example, melt-through could occur on the next cycle to T_C even though the liquidus temperature for the shot as a whole remained T_B .

If the highest temperature in the cycle were restricted to being just above the eutectic temperature, it would be possible to melt the eutectic core with little change in the average thickness of the silicon shell. In the concept, however, the stored energy density is increased by cycling to temperatures about midway between T_{eut} and T_B , the dissolution and redeposition of a portion of the silicon shell thus contributing a corresponding latent heat of fusion of silicon.

Mobley and Rapp (1983) (Ref. 2) proposed, and investigated, a number of means to form integral silicon shells. These means included: (1) controlled quenching of molten spheres of aluminum-silicon alloy, having hypereutectic composition, to produce integral shells of the primary precipitate of Si; (2) aluminum-silicon alloy, having hypereutectic composition, to produce integral shells of the primary precipitate of Si; (2) aluminothermic reaction coating, in which liquid Al-Si alloy is allowed to react with solid SiO_2 to form a shell of solid Al_2O_3 (alumina) and silicon; (3) packed bed cementation siliconizing, in which silicon is deposited on the surface of solid alloy shot via vapor transport from a bed of silica and a mixed chloride salt. Although the authors reported that the pack-cementation coating method seemed to show

promise, no consistent results were produced that showed the formation of encapsulated spheres which did not crack or leak via exudation of alloy through the Si shell.

The first question to be answered is: does an integral shell of Si, produced by whatever means on eutectic Al-Si shot, remain stable with repeated thermal cycling around the eutectic temperature (and of course below the liquidus temperature)? For, even with an integral shell of Si around the Al-Si eutectic, and with a constant temperature, there still is a driving force for recrystallization of Al and Si, and of crystal growth, which could lead to growth of Si crystals inward and of Al crystals outward. This could lead to thinning of the shell, pinholes and leaks, or collapse of the shot. A rough first estimate of the probability of this occurring would require estimates of the diffusion coefficient. For example, Darken and Gurry³ consider the case of diffusion into (or out of) variously shaped objects, initially of uniform composition, whose surface is in contact with a source of different composition. The fractional saturation (i.e., the amount that has diffused into the object relative to the amount that will have entered at infinite time) is a function of ℓ/\sqrt{Dt} . For a slab, or sheet, the diffusion process is 93% complete when $Dt/\ell^2 = 1$. Diffusion is 36% complete when $Dt/\ell^2 = 0.1$, i.e., for a tenfold smaller diffusion coefficient. Thus, the relaxation time for a diffusion layer is $\tau \sim \ell^2/D$ where ℓ is the half-thickness of the layer and D is the diffusion coefficient. For relaxation in, say, 3 h ($\sim 10^4$ s), of a layer of thickness $\sim 10^{-2}$ cm, the diffusion coefficient would have to be $D \approx 10^{-4}/10^4 = 10^{-8}$ cm²s⁻¹. This value may appear somewhat high for solid state diffusion (in the liquid state $D \sim 10^{-5}$ cm²s⁻¹; for many ceramic solids, $D \sim 10^{-12}$ cm²s⁻¹). However, some alloys, e.g., Li-Al, Li-Si, do have diffusion coefficients as high as 10^{-8} cm²s⁻¹ or higher. A diffusion coefficient of this magnitude could lead to deterioration of the shell on cycling unless there is some constrain to block it, such as a temperature gradient of appropriate direction and magnitude. Mondolfi⁴ reports relatively high diffusion coefficients in solid Al, and high diffusional mobility in the grain boundaries. Rhines and Aballe⁵ have observed Ostwald ripening and growth of silicon crystals in solid

silicon-aluminum alloys at temperatures as low as 540°C. The diffusion coefficients in the liquid are higher still.

A perhaps more fundamental factor is the stability of the silicon shell to redistribution during repeated melting and crystallization, which could lead to disintegration of the shell. A number of mechanisms could provide the driving force for such redistribution. These include:

1. a small temperature gradient in a fixed direction across a shot that provides a thermodynamic driving force for preferential deposition of Si crystals on the cool side, leading to thinning of the shell on the hot side.
2. dendritic growth of primary silicon crystals from supercooled liquid leading to entrapment of alloy leading to migration of aluminum from the interior of the shot to the surface.
3. relatively high diffusion coefficient of silicon in aluminum providing a mechanism, even isothermally in the solid state, for interdiffusion of silicon and aluminum, with gradual redistribution of silicon and aluminum.

Thus, although the equilibrium relationships show the possibility of an integral silicon shell, the equilibrium is dynamic, not static; hence the distribution of the silicon and aluminum phases must be considered.

As part of a test of the integrity of silicon shells to be used as containers for Al-Si eutectic, experiments were designed to investigate the changes in shape of the interface between silicon and aluminum-silicon eutectic alloy on repeated melting and freezing of the alloy in contact with silicon. The experiments were intended to be independent of methods used to fabricate self-encapsulated shot, and to provide basic information concerning the inherent stability of such shells to migration via crystal growth and ripening under conditions of temperature cycling. The basis of the experiments was the preparation of polycrystalline shapes such as bars of silicon to be heated in contact with Al-Si eutectic, maintained at temperatures between 550 and 650°C for varying lengths of time, cooled, and then subjected to optical and metallographic examination for dimensional stability. Two sets of

experiments were conducted: (1) the temperature of the samples was cycled between 550 and 650°C, and (2) the samples were held for differing periods of time at various constant temperatures above the eutectic temperature (577°C) before examination. The latter, isothermal, experiments provided an indication of the inherent rate of redistribution of silicon and alloy resulting from the dynamic equilibrium even in the absence of a significant temperature gradient, e.g., via Ostwald ripening. The former, temperature cycling, experiments were more complex in that some of the solid silicon must dissolve in the liquid alloy during the heating part of the cycle, and then recrystallize on the Si surface during the cooling part of the cycle. The temperature cycling conditions simulated more closely the behavior of a silicon shell during cycling of encapsulated shot, although the stresses induced by volume expansion may be smaller than in spherical shot.

The experiments to date have shown that initially flat surfaces of contact between Si and Al-Si eutectic show development of a lenticular shape after heating for several hours at temperatures above the eutectic temperature (577°C). The penetration is very slight for temperatures up to 600°C; in some cases, possibly because of oxide films, there is no evidence of wetting. At 650°C and above interpenetration generally occurs. At 700°C and above, the interpenetration occurs to such a large extent that further experiments at these temperatures were discontinued, and subsequent experiments were done at 650°C.

In these subsequent experiments, two variations of the penetration were observed. One was the formation of interpenetrating "fingers" of Si and Al-Si, and "roughening" of the interface. This appeared in most cases, at 650°C, to be of the order of 0.15–0.25 mm. The interfacial roughness which did not appear to progress markedly with increased heating time or with cycling, would suggest that shells of the order of 1 mm thickness could be stable to cycling up to 650°C. However, the initially proposed shell thickness of 0.1 mm would clearly not be stable. Stress cracks were found to occur at distances less than 1 mm from the interface. This suggests that thick shells to mitigate interpenetration of the Al-Si may be mechanically weak.

The second modification of the lenticular penetration was the intrusion of nearly spherical globules of eutectic, of the order of 2-3 mm in diam, into the silicon. There was no evidence that this intrusion occurs because of a preexisting void of that size in the silicon. Although attempts were made to determine whether such intrusions occurred preferentially at scratches or slots inscribed in the Si substrate, intrusion did not occur preferentially at those sites. Such intrusions, which occurred with about 10% of the samples heated to 650°C, could eliminate the possibility of stable self-encapsulated shot, and the mechanism of formation of these intrusions would have to be established.

In summary, application of self-encapsulated Al-Si shot was found not feasible for temperatures of 700°C and above. It may be feasible for temperatures up to 650°C if the mechanism of globular intrusion can be determined and controlled, and if temperature excursions to 700°C can be avoided. Application at temperatures up to 600°C appears feasible if a somewhat reduced storage capacity is acceptable and if temperature excursions beyond 600°C can be avoided.

2.3 Heats of Mixing for TES Systems (WBS 1.3.1.1)

Liquid-liquid systems can be of several types depending on the molecular interactions which cause the limited miscibility. If the solubility of the components in each phase increases with increasing temperature, an upper critical solution temperature (UCST) is reached above which the components are miscible in all proportions. This behavior is shown in Fig. 5 for the aniline-n-heptane system.⁶

For some systems, such as water-amines, the region of limited miscibility occurs only above a lower critical solution temperature (LCST). Systems of this type may also have upper critical solution temperatures above which the components are completely miscible, as shown in Fig. 6 for glycerol-m-toluidine.⁶ For these systems strong polar interactions account for the complete miscibility at low temperatures, while at intermediate temperatures the dissimilarity of the molecules

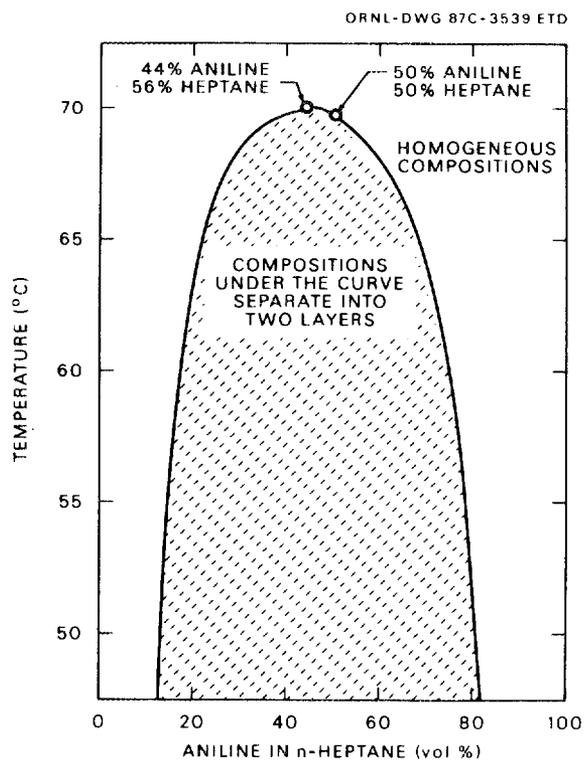


Fig. 5. Liquid-liquid phase behavior for aniline-n-heptane system.

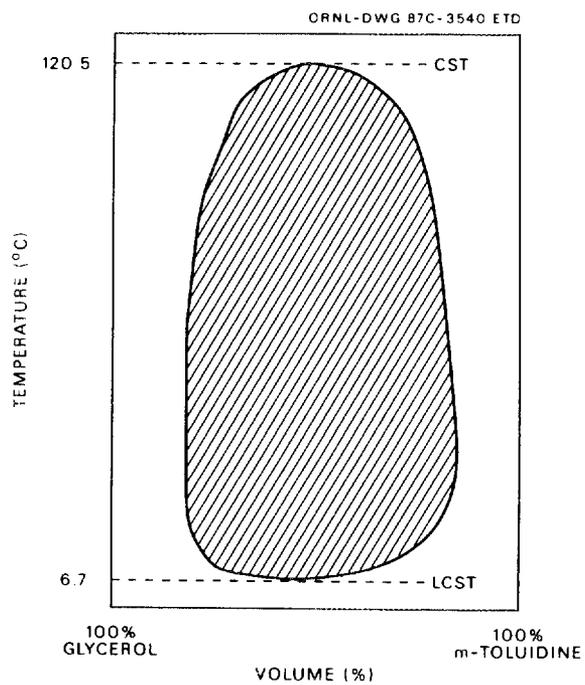


Fig. 6. Liquid-liquid phase behavior for glycerol-m-toluidine system.

causes the phase separation. At high temperatures the dissimilarity in polarity effects are reduced and the components are again miscible.

The heat of mixing is positive at the critical composition, x_{1C} , for a system with an UCST, while it is negative at a lower critical point. Therefore, for systems with both lower and upper critical points the heat of mixing must vary from negative to positive with increasing temperature, and be zero at an intermediate condition.

For liquid-liquid systems, the heat of mixing varies linearly with composition over the composition range of immiscibility. This is because for these compositions the heat of mixing for the two phase system, H^E , is determined only by the relative amounts of the coexisting liquid phases. This behavior is illustrated in Fig. 7 for the

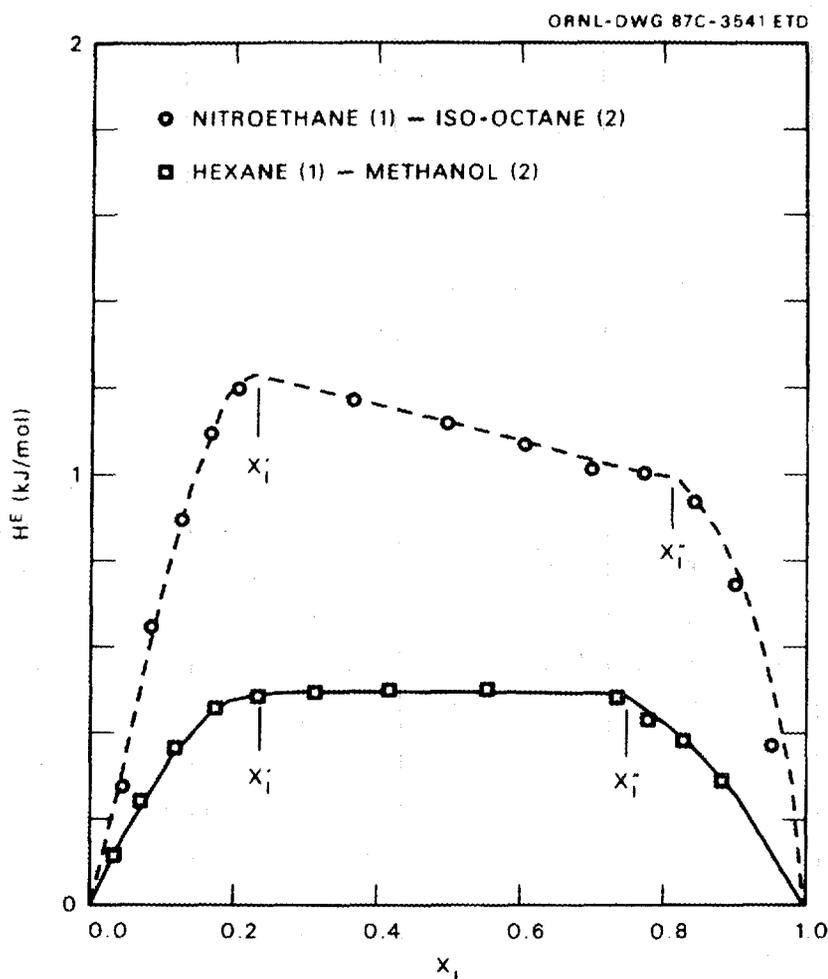


Fig. 7. Heats of mixing for nitro-ethane-iso-octane and hexane-methanol system below their CST's.

nitroethane-iso-octane and hexane-methanol systems for temperatures below their upper critical values.

The heat of mixing H^E at an overall mole fraction, x_1 , between those for the coexisting phases, x_1' and x_1'' , can be represented as

$$H^E = a + bx_1 \quad (1)$$

where

$$a = \frac{x_1' H^{E''} - x_1'' H^{E'}}{x_1' - x_1''} \quad (2)$$

$$b = \frac{H^{E'} - H^{E''}}{x_1' - x_1''} \quad (3)$$

and $H^{E'}$ and $H^{E''}$ are the heats of mixing for the coexisting phases.

The enthalpy of a liquid mixture with a composition x_1 is

$$H = x_1 H_1 + x_2 H_2 + H^E \quad (4)$$

From Eq. (4), the enthalpy difference between two temperatures T_2 and T_1 , above and below an upper critical solution temperature, is given by

$$\Delta H = \bar{C}_p^0 (T_2 - T_1) + H_{T_2}^E - H_{T_1}^E \quad (5)$$

where $\bar{C}_p^0 = x_1 C_{p1}^0 + x_2 C_{p2}^0$, C_{p1}^0 and C_{p2}^0 are average heat capacities of the components, and $H_{T_1}^E$ is obtained from Eq. (1) for compositions between x_1' and x_1'' . Equation (5) also represents the enthalpy difference for temperatures above and below a LCST, with H^E obtained from Eq. (1) for compositions in the two liquid phase region.

For effective energy storage, large differences in enthalpy, and therefore in $H_{T_2}^E - H_{T_1}^E$ are required for small temperature differences $T_2 - T_1$. Equation (5) can be expressed in the alternate form

$$\Delta H = \bar{C}_p M(T_2 - T_1) \quad (6)$$

where \bar{C}_p and M are the mean heat capacity (J/g) and molecular weight of the mixture. From Eqs. (5) and (6),

$$\bar{C}_p = \frac{\bar{C}_p^o}{M} + \frac{H_{T_2}^E - H_{T_1}^E}{M(T_2 - T_1)} \quad (7)$$

The last term on the right side of Eq. (7) indicates the contribution of the heat of mixing to the effective system heat capacity.

Experiments by others⁷ have shown the heat of mixing to increase the effective heat capacities of some chemical systems by 100% over a small temperature range.

For heat storage, a mixture with an UCST is heated from the two liquid phase region to above the critical point to form a single phase solution. For a system with a LCST, the heating process results in a transformation from a single phase to two liquid phases. Heat is then provided to the building space by cooling the solution to the original condition. The system can also exhibit a solid-liquid phase change at different conditions to aid in the storage capabilities.

For cool storage, an aqueous system can be used with a critical solution temperature of $\sim 5^\circ\text{C}$. The mixture is cooled from 10°C to below its critical solution temperature. The cool energy can be recovered by heating the mixture to its original condition.

Studies were performed to find potential systems with UCST'S in the range $40^\circ\text{--}70^\circ\text{C}$ for heat storage applications. Aqueous mixtures containing organic components with critical solution temperatures in this range can also be used for combined heat and cool storage. For cool storage the system would form a solution of ice and the liquid component at about 0°C . Optimum systems of this type would give large changes in the heat of mixing in the critical region. Several nonaqueous systems with this property have been identified, including aniline-n-hexane and cyclohexane acetic acid anhydride. From available experimental data,^{9,10} these systems exhibit maximum heats of mixing greater than 3000 J/mole.

In Figs. 8 and 9 the experimental heats of mixing for these systems are plotted against composition. In Fig. 8 for aniline-n-hexane, the

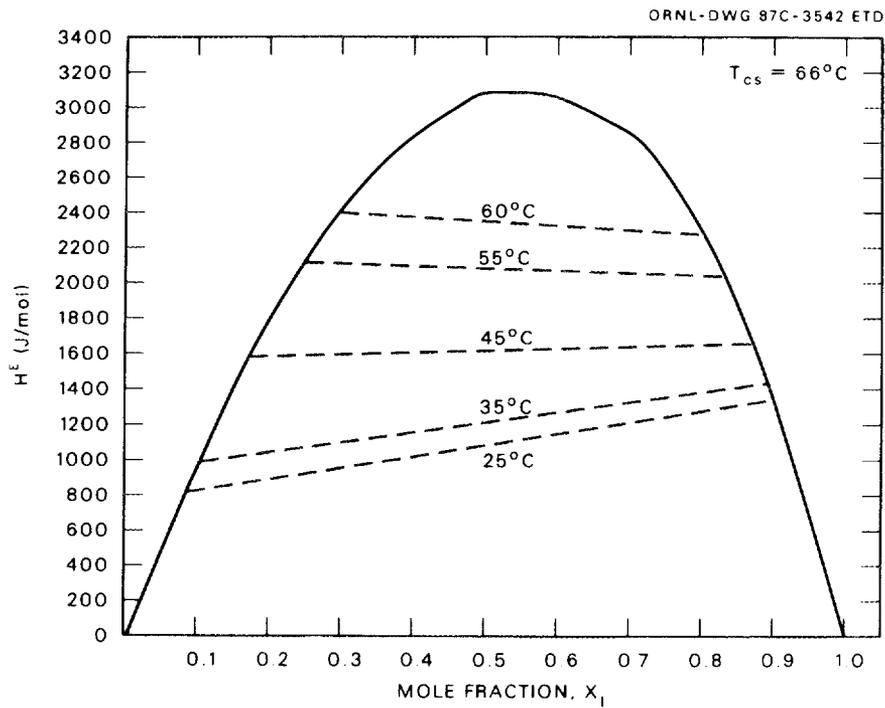


Fig. 8. Experimental heats of mixing for aniline (1)-hexane (2).

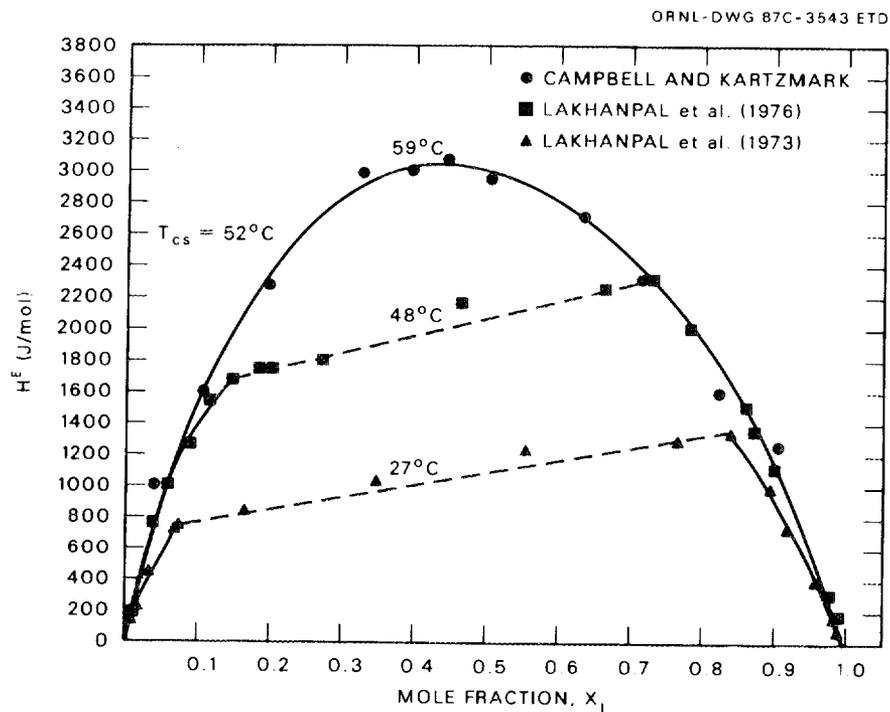


Fig. 9. Experimental heats of mixing for acetic acid anhydride (1)-cyclohexane (2).

approximate values of H^E at the phase boundaries for temperatures below the UCST's are also indicated. For aniline-n-hexane in the region 60–66°C, the increase in the heat capacity for equimolar mixtures indicated by Eqs. (6) and (7) is ~ 1.7 J/g°C. The effective heat capacity of the liquid-liquid mixtures is almost double that of the homogeneous liquid mixture. An aqueous mixture with a similar heat effect would have a large increase in the heat capacity because of the lower molecular weight of the system.

The propylene carbonate-water system (UCST = 60–70°C) has been proposed as a potential system for heat and cool energy storage applications. Other potential aqueous mixtures with UCST's in this range are listed in Table 2.

Table 2. Aqueous systems

	UCST (°C)
o-aldehydrobenzoic acid	45.7
m-aminobenzoic acid	66.0
p-aminobenzoic acid	47.0
Benzamide	<75
Glutaric dinitrile	68.3
m-hydroxybenzaldehyde	66.2
p-hydroxybenzaldehyde	64.4
o-nitrobenzoic acid	52
Novocaine thiocynate	54
Phenol	66
o-phenylenediamine	<67
p-phenylenediamine	<75
Phenylhydrazine	55.2–75.5
Piperonal	<78
Succinonitrile	54
Trichloroacrylic acid	60.85

Since heat of mixing data are not available for these aqueous liquid-liquid systems, in this study the use of activity coefficient models to estimate H^E from the phase solubility data was investigated. Initial calculations were performed by the use of experimental data for

the phenol-water,¹¹ methanol-hexane,¹² and methanol-heptane¹² systems with procedures based on the Van Laar, Margules, Uniquac and NRTL models.

For systems with lower critical solution temperatures, a more complex relationship for the temperature variation of the constants in each model are required, since the heat of mixing can vary from negative to positive in the two liquid phase region. For several aqueous systems with LCST's, including water-2-butanone, water-3-methyl pyridine, and water-2-6-dimethyl pyridine, the Uniquac procedure, was found to result in close agreement with the experimental values of H^E . Good agreement with the experimental heats of mixing also resulted for the Uniquac procedure for the water-2-4 dimethyl pyridine system. Larger errors resulted with the same phase data by the use of the Van Laar model. These results indicate that accurate heats of mixing can be estimated for aqueous systems with LCST's by the Uniquac procedure if highly accurate phase data are available for a suitable temperature range. If sufficient liquid-liquid phase data are not available, values of H^E can also be estimated for these systems for accurate vapor-liquid equilibrium data in the single liquid phase region.

The results of this study indicate that the aqueous systems considered with UCST's have moderate increases in the effective heat capacities in the critical regions of these systems: experimental studies are required to establish the exact thermal energy storage capabilities.

A number of systems with LCST's of $\sim 50^\circ\text{C}$ have been identified which appear to show large differences in the heat of mixing in the critical region. Systems with this behavior are promising for potential heat and cool storage applications and include mixtures of water with methyl diethyl amine, 2-6-dimethyl pyridine, dimethyl-tert-butyl amine, and 3-methyl pyridine. Experimental studies will be conducted in Phase II of this investigation for several of these mixtures with a flow calorimeter system to confirm the predicted behavior.

Aqueous systems with liquid-liquid behavior in the range 0 to 10°C may also have potential for cool storage applications. Several potential pyridine and amine systems with lower critical solution temperatures in this range are listed in Table 3. These systems will also be investigated further in Phase II of the Program.

Table 3. Potential aqueous mixtures for cool storage

Component	LCST (°C)
2,4,6 trimethylpyridine	3.5
3,4 dimethylpyridine	-3.6
Dipropyl amine	-4.9
1-ethylpiperidine	7.45
2-ethylpiperidine	-5.0
3-ethyl, 4-methylpyridine	6.4

2.4 Medium Temperature Composite Media (WBS 1.3.1.2)

Development of a medium temperature composite thermal energy storage material was begun under WBS project 1.3.1.2. The goal was to define a set of materials that could store latent heat energy in the 500°F to 950°F temperature range. The justification for this work is that a packed bed TES unit with only one PCM (i.e., one melt temperature) has little additional storage capacity (and therefore little economic advantage) over a traditional sensible heat storage system if large ΔT 's are permissible. This limitation had been recognized for some time, but the development of the high temperature $\text{Na}_2\text{CO}_3\text{-BaCO}_3/\text{MgO}$ composite had only recently matured to the point where it was appropriate to begin developing a family of composite materials. During the previous year, two material combinations were identified, fabricated and partially characterized.¹³ Further development was postponed pending the completion of design optimization studies. These studies were needed

either to confirm the 500°F to 950°F operating range, or to indicate a more appropriate temperature range.

Using evaluation criteria such as heats of fusion, relatively low material cost, appropriate melting point, material stability, and environmental safety; three candidate PCM's were chosen for initial testing. These were boron oxide, sodium nitrate, and a ternary eutectic carbonate of sodium, lithium, and potassium. Through a subsequent evaluation, it was learned that boron oxide is usually manufactured from boric acid and the resulting oxide is in the amorphous state. This was deemed undesirable because meaningful energy storage only occurs when the crystalline form melts. Even though manufacturing techniques and dopants were available which would force the crystalline state to form and remain stable, it was felt that these additional steps would increase manufacturing costs to the point where the boron oxide would no longer be cost effective. The remaining candidate, sodium nitrate and eutectic carbonate PCM's were subjected to material compatibility studies. For this initial series of tests, the sodium nitrate and eutectic carbonate PCM's were dry milled and pressed into 0.6 cm (1/2-in.) diam cylindrically shaped pellets. As with the high temperature composite media, Magnesium Oxide (MgO) was used as the substrate material. A significant number of each were then subjected to densification and weight loss studies.

Two batches of pellets were fabricated using the sodium nitrate salt. Each batch contained pellets with either 50%, 60%, or 70% salt volumes. One batch was sintered at 425°C and one at 525°C. In each case, the pellets achieved a final theoretical density which was a function of their salt content. Those sintered at the lower temperature achieved 63%, 70%, and 77% of theoretical density for the 50%, 60%, and 70% salt volumes respectively. Those sintered at the higher temperature achieved 68%, 72%, and 89% of theoretical densities respectively. A single batch of pellets was fabricated using the eutectic carbonate, with individual pellets containing either 50% or 60% salt volume. This batch was sintered at between 900–950°C and achieved between 80 and 82% of theoretical density. These densification results were considered only marginally acceptable (92% of theoretical density being routinely

achieved for the high temperature components), but sufficient to permit the compatibility studies.

The goal of the studies was to evaluate composite pellet stability, PCM (i.e., salt) retention characteristics, composite system (i.e., combined salt and substrate) stability, and temperature limitations of the system. The studies were accomplished by cycling a given batch of pellets through their melting point and soaking (i.e., holding at a constant temperature) for 20 h. After each cycle, the pellets were cooled to room temperature and weighed. This thermal cycling/density measurement process was repeated until a significant amount of total time above the melting point had been accumulated.

Compatibility test results are summarized in Figs. 10, 11, and 12. Each figure shows the weight loss of a specific PCM as a function of total accumulated time above the melt point. Figure 10 shows the weight loss data for the sodium nitrate PCM that had been sintered at 425°C and soaked at 400°C. These results show that no measurable weight

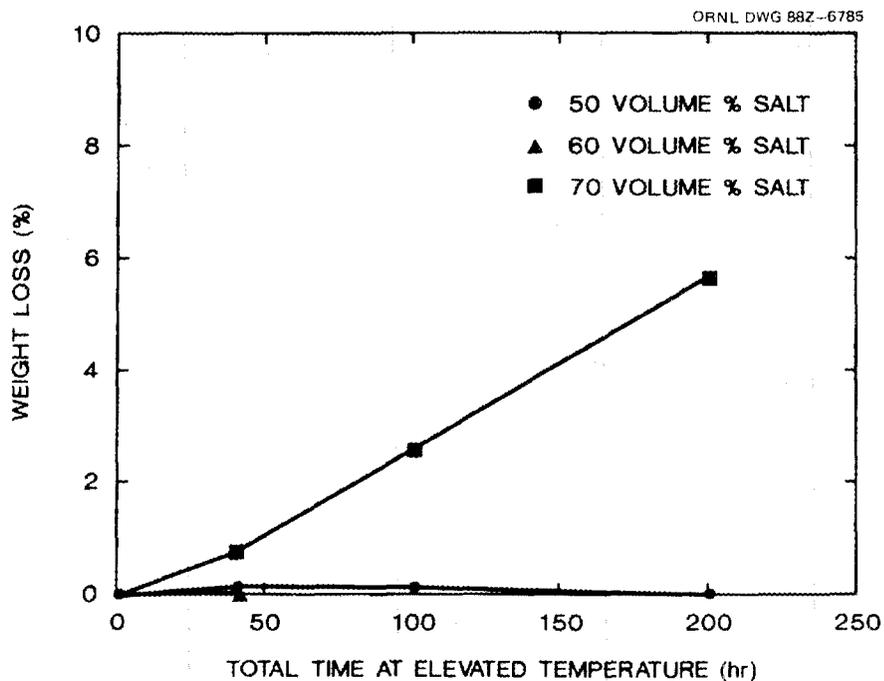


Fig. 10. Weight loss of NaNO_3/MgO pellets sintered at 425°C; soaked at 400°C.

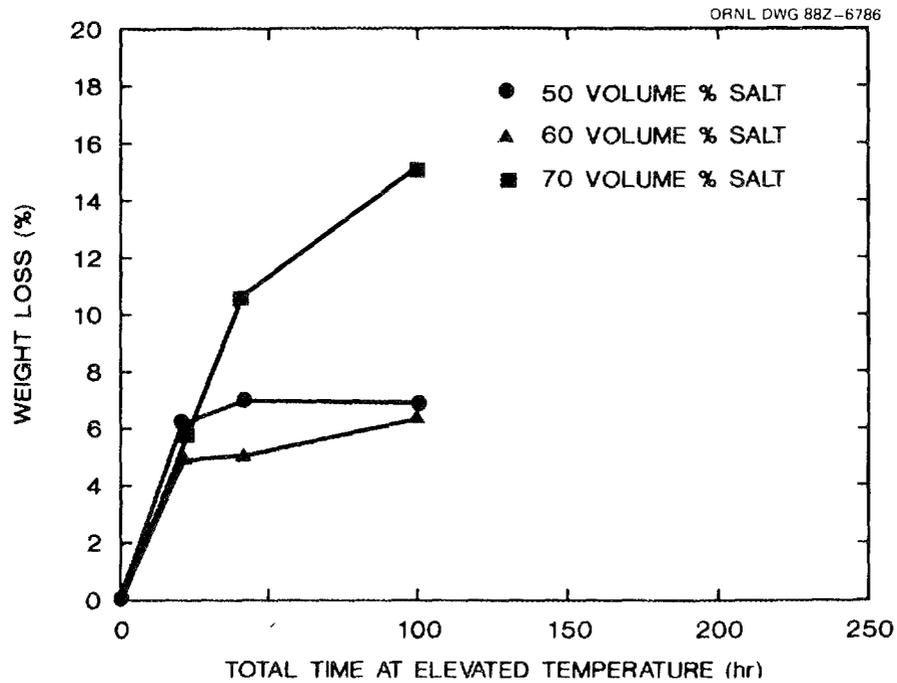


Fig. 11. Weight loss of NaNO₃/MgO pellets sintered at 525°C; soaked at 400°C.

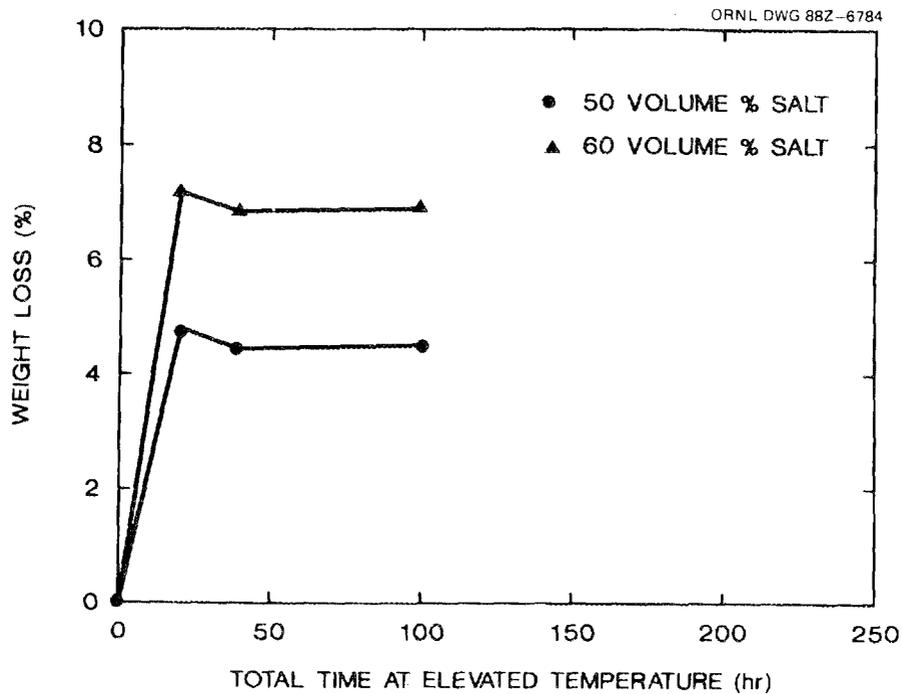


Fig. 12. Weight loss of Na, Li, K ternary eutectic pellets sintered at 900–950°C and soaked at 500°C.

loss occurred in the samples containing 50% and 60% salt volumes. However a significant weight loss occurred in those pellets with 70% salt volume. These data suggest (for those sintered at 425°C) that the sodium nitrate salt content should be kept at or below 60% to maximize storage benefits while avoiding salt losses.

Figure 11 shows the weight loss data for the sodium nitrate pellets that had been sintered at 525°C. These results show that all pellets experienced significant salt loss during the first cycle. Subsequent cycles resulted in little additional loss for the 50% volume pellets, slightly more loss for the 60% volume pellets and considerable additional loss for the 70% volume pellets. It appears that the 50 and 60% data converge to an asymptotic value of 6% weight loss. Figure 12 shows the test results for the ternary eutectic carbonate that had been sintered at 900 to 950°C. These data indicate that a significant amount of salt was lost during the first cycle, but very little thereafter. It was not possible to draw conclusions concerning the long-term stability of the two medium temperatures composites based on these data. Further materials selection and testing were temporarily postponed pending the completion of system design optimization studies.

2.5 Assessment of Regulations on Ammoniate Cycles

The release of chlorofluorocarbons into the atmosphere and potential destruction of the earth's ozone layer is an environmental issue of growing concern. Indications are that stringent production ceilings will be placed on the manufacture of most of the refrigerants (R-11, R-12, R-113, R-114, R-115) now used in building air conditioning systems so that refrigerant alternatives will be required. One alternative may be ammonia (R-717) used as a working fluid in new refrigeration systems. Of the typical refrigerants, ammonia is an excellent working fluid. Its heat of evaporation is very high, second only to that of water, and its saturation pressures at temperatures needed for cooling systems can be controlled without difficulty using existing technologies. On the other hand, since ammonia is combustible, corrosive and toxic, applications may be limited. Based on the thermodynamic potential of thermal storage systems using ammonia, studies were completed

to assess the regulatory issues associated with ammonia storage and use in buildings. The following documents reviewed represent a cross section of those used throughout the U.S.:

1. Uniform Codes of the International Conference of Building Officials (ICBO) which are used as a basis for most codes west of the Mississippi River and the State of Indiana,
2. Basic/National codes of the Building Officials and Code Administrators International, Inc. (BOCA) which are used as a basis for most codes east of the Mississippi River, north from Kentucky and Virginia and including parts of Oklahoma and Texas,
3. Standard codes of the Southern Building Code Congress International, Inc. (SBCCI) which are used for most codes east of the Mississippi River, south from Tennessee and North Carolina and including parts of Texas,
4. State-developed codes of North Carolina and Wisconsin,
5. Major city codes of New York, Chicago, and Los Angeles,
6. National Fire Protection Associations standards which are referenced in the above documents as well as being adopted directly by many state and local regulatory agencies,
7. Ammonia Safety Requirements of the American National Standards Institute (ANSI),
8. Mechanical refrigeration safety codes by the American Society of Heating, Refrigerating, and Air-Conditioning Engineers, Inc. (ASHRAE).

Since current building regulatory criteria were found to vary significantly across the U. S., a simple, uniformly-applicable set of conclusions regarding the use of ammonia in buildings could not be drawn. On a worst case basis, an ammoniated storage system design was unacceptable. At best, the system is acceptable for any applications as long as the pressure vessel is ASME approved, has proper clearances and is properly vented.

2.6 Thermal Energy Storage With Clathrates

Current cool storage technologies are limited either in thermodynamic efficiency or are quite large in size if based on sensible cooling. Ice storage systems, for example, have relatively high storage densities [88.3 cal/g (159 Btu/lb) when operated over a 8.3°C (15°F) temperature range] but are somewhat disadvantaged in that low evaporator temperatures (and resulting low refrigerating efficiencies) are needed; cool storage systems based on chilled water storage have low energy densities [\sim 8.3 cal/g (15 Btu/lb)] but are relatively efficient since the evaporator is simply a water chiller operating over an approximate temperature range of 8.3°C. In prior efforts,^{14,15} refrigerant clathrates as "warm ices" were examined experimentally as an alternative to both ice and chilled water since they effect latent cool storage at temperatures higher than ice. As a cool storage medium, clathrates appear to be advantaged over ice and chilled water. A study to evaluate the technical advantages of clathrates as demonstrated through experimental evidence and issues that disadvantage clathrate cool storage concepts was initiated and completed. With direct contact evaporators, clathrate studies indicate that compressor saturated suction temperatures are approximately that of the clathrate critical point (e.g. 12°C for R-12). The estimated improvement in theoretical efficiency of a direct contact R-12 clathrate system over the conventional ice-on-coil technology is \sim 40% based on a 11°C higher evaporator temperature as available with the clathrate system. This advantage justified prior research efforts by private industry and the DOE to investigate cool storage concepts using clathrate technology.

An evaluation of clathrate technologies was completed which indicated that despite performance improvements, significant cost and environmental issues remain unresolved with clathrate cool storage systems. The cost of the refrigerant inventory in an ideal clathrate composition of R-11 and water and at 100% conversion is \sim \$4.80/kWh. This is a significant additional cost if judged against the \$14/kWh total cost of ice storage. The presence of refrigerant gas as part of the storage system, requires a vessel suitable for containment of the

clathrate under pressure or vacuum, further adding to the cost of a clathrate cool storage system. Of still greater consideration are the environmental concerns with systems that require large inventories of chlorofluorocarbons. Production ceilings for R-11 and R-12 which proved to be the most practical clathrate refrigerants will no doubt hamper further development of cool storage systems based on clathrates.

2.7 High Temperature Composite Media Economic Assessment (WBS 2.3.4.1)

An economic assessment of the composite media was conducted by ORNL as a part of the TES Program decision making process. Its purpose was to determine if the composite storage media retained an economic advantage over comparable sensible heat TES systems, and to indicate appropriate research and development activities for the coming fiscal year.

Current financial and technical information was used to update a preliminary benefit analysis which had been conducted in CY-83 by the Institute of Gas Technology (IGT). Both analyses utilized a life cycle based, value analysis methodology which employed the following four step evaluation technique:

- (1) The value of recovered heat was determined by calculating the present worth of the annual net energy savings associated with the operation of a TES system. This savings was defined as the algebraic sum of the kiln fuel (e.g., natural gas) savings, TES annual O&M costs, and parasitic power requirements (i.e., kiln fan power).
- (2) A maximum allowable TES system capital cost was defined as the present worth of the annual net energy savings.
- (3) The capital costs for different sensible and latent heat TES system designs were estimated.
- (4) The estimated capital cost for each TES design was then compared to the maximum allowable capital cost to see if it was cost effective; that is if its cost was less than the maximum allowable.

The analysis conducted during CY-83 utilized a simplified set of operating and financial assumptions. Plant process assumptions included: a kiln burner efficiency of 85%, one TES charge/discharge cycle per day, 300 operating days a year, and temperature swings of $\sim 450^{\circ}\text{F}$. The financial assumptions included a natural gas price of \$5.75 per million Btu's, and a capital cost that was the sum of the individual costs for the storage media, media containment, and flue gas distribution system. The results of this preliminary study are summarized in Fig. 13 and show the composite storage media to be superior to conventional sensible heat storage systems.

The updated analysis utilized the results of recent IGT research activities and improved on the initial set of assumptions in three areas:

1. The price of natural gas was reduced to \$5.00 per million Btu's to reflect the current energy market.
2. A range of composite storage media costs were defined that reflected fabrication experience gained during recent research and development. These were an extrusion process and a dry milling process

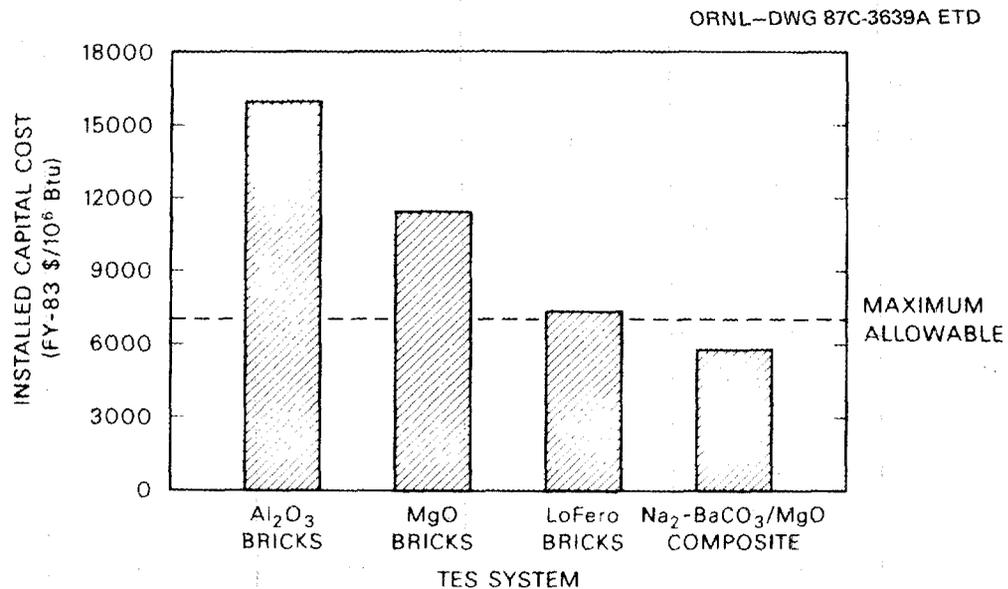


Fig. 13. Economic comparison of sensible heat systems and high temperature composite medium.

that produced pellets with unit prices of \$0.30 and \$0.19/lb, respectively.

3. The use of different sized spheres of composite media to increase the packing density and thus the total amount of PCM available for latent heat storage.
4. A more realistic set of process assumptions. A careful study of the brick industry revealed several operating characteristics that impacted the design (and therefore the capital costs) of TES systems. These were:
 - a. Existing brick industry heat recovery systems operate below 950°F to avoid using stainless steel as a material of construction.
 - b. Installation of TES systems serving multiple kilns and dryers results in an almost constant heat demand and rejection. This in turn permits very short storage/removal periods.

Several individual studies were conducted to determine the impact of each of these considerations on the economic viability of the composite storage media. The first was an assessment to show the effect of increased packing densities and different media costs for two types of TES systems; one that operates below 950°F and one that operates above 950°F. A second assessment was performed to determine the effect of the number of TES cycles per day on the installed capital cost of a latent heat TES system. Capital costs for both sensible and latent heat storage system as a function of the number of TES cycles per day were compared for both the low and high temperature systems. The effect of different media cost and packing densities for the high temperature system are shown in Fig. 14.

The assumptions included (a) stainless steel as a material of containment construction, (b) temperature swings of 900°F, and (c) a $\text{Na}_2\text{CO}_3\text{-BaCO}_3/\text{MgO}$ composite storage media. Figure 14 which compares capital costs for the fabricated media of 19 and 30¢/lb and void volumes of 25% and 40%, indicates that all price and density combinations, except the most expensive/least dense, appear economically viable. A similar analysis was performed for the low temperature media with a temperature swing of 450°F and mild steel for the TES unit and

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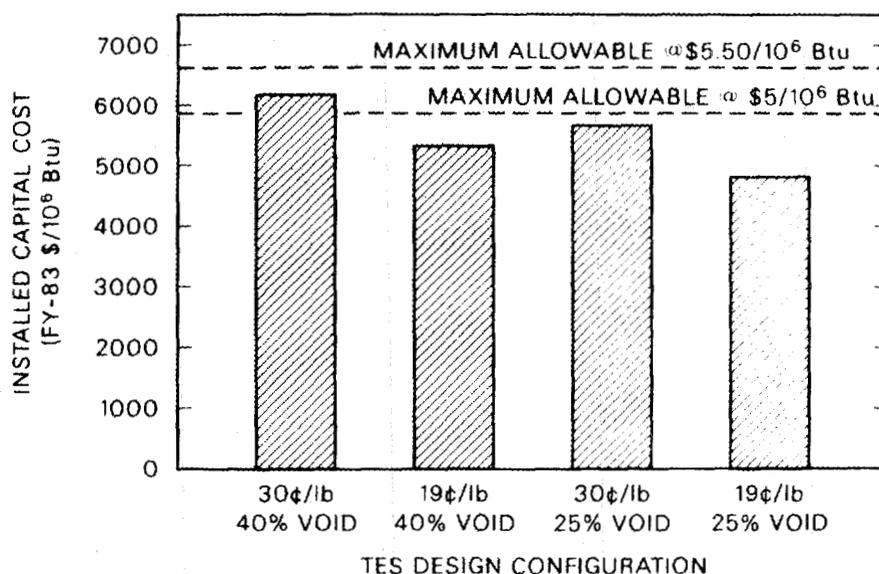


Fig. 14. Effect of $\text{Na}_2\text{CO}_3\text{BaCO}_3/\text{MgO}$ (high temperature) composite media cost and packing density on system installed cost.

distribution system. The results of this analysis, shown in Fig. 15 are very similar to those for the high temperature systems (Fig. 14). The installed cost of a smaller, stainless steel container for the high temperature media is approximately equal to the installed cost of a larger mild steel container for the low temperature medium.

To determine the effect of the number of cycles per day on the economic viability of latent heat TES systems, two separate calculations were made. These were one to determine the effect of number of cycles on the installed capital cost of a high temperature, latent heat TES system, and a second to compare sensible and latent heat systems capital costs. Figure 16 summarizes the effect of increasing number of cycles per day on the installed capital cost of a nominal high temperature system. The assumptions were a constant total daily heat load and a storage unit that utilized storage media purchased at 30¢/lb and packed with a 25% void volume. As expected, the costs decrease as the number of cycles increases. This is because as the number of cycles per day increases, progressively smaller units can be built to deliver the same total heat load.

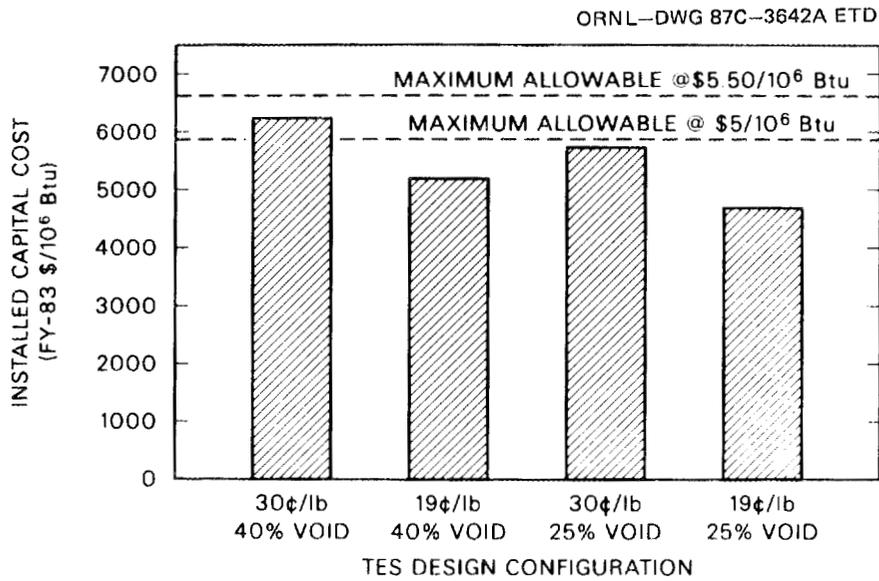


Fig. 15. Effect of NaNO_3/MgO (low temperature) composite media cost and packing density on system installed cost.

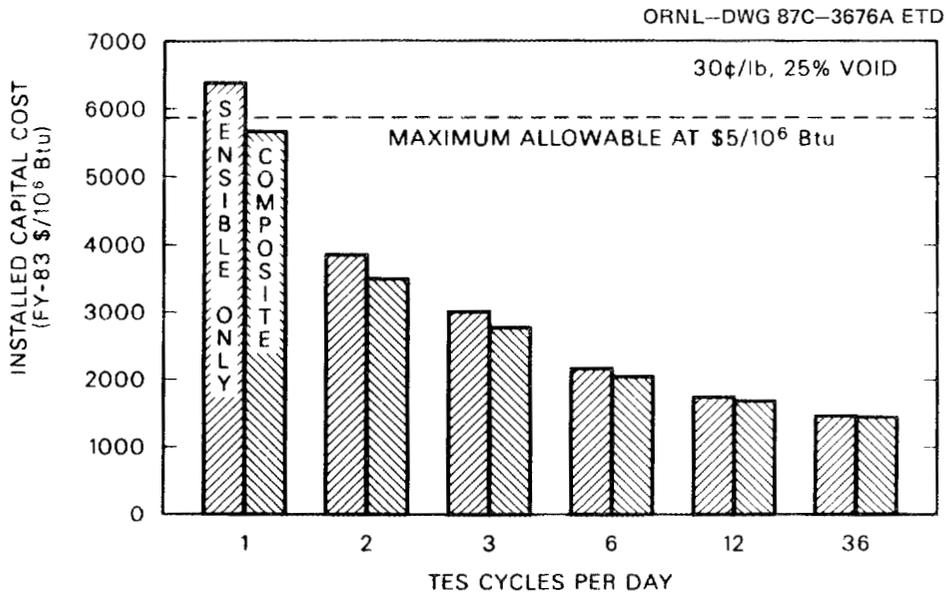


Fig. 16. Effect of storage utilization on installed cost of high temperature system.

An interesting point is that the cost difference between a sensible and latent heat system (for the same number of cycles) also decreases for increasing cycles. The difference is due to the volume savings that result when latent heat storage media are used instead of sensible heat media. The difference decreases as the size of the unit decreases because the portion of total costs attributable to the storage media and containment decreases. Thus any savings in storage unit volume which result from using a latent heat media become less significant.

Based on these results, the following conclusions were reached:

1. the composite media can be cost effective in certain system designs for energy recovery in a brick plant,
2. packing densities greater than 60% will be required depending on the purchase price of the composite storage media,
3. the composite media has an economic advantage over traditional sensible heat systems but the extent of this advantage is a function of system design and operation,
4. there is sufficient potential to justify continued development of the composite storage media, and
5. future economic analyses should be conducted using a systems analysis approach; that is an optimization design procedure. This will insure that all system technical and cost considerations will be used to maximize the economic benefit for each storage configuration. This is especially critical since the existing assessments indicate a range of system designs where the composite media does not have a clear economic advantage.

2.8. High Temperature Industrial TES Project

During the reporting period, a comprehensive plan for the development, testing, and evaluation of phase change energy storage materials for industrial applications was defined and implemented. Isolated research and development (R&D) activities were redirected under the plan's control to insure that a consistent decision making

methodology could be used to evaluate results and determine future research directions. There were four main reasons for this redirection; (1) the adoption of systems analysis techniques as the primary decision making tool to determine the technical and economic viability of TES storage media and/or systems, (2) the decision to initiate R&D on a medium temperature composite storage media, (3) the addition of Pacific Northwest Laboratories (PNL) to the Industrial TES project, and (4) the decision to proceed with the next phase of the high temperature storage media development, a subscale test facility in an operating brick plant. To incorporate these new constraints in a logical, orderly and controlled fashion, it was necessary first to define a most general project execution sequence and then execute the sequence for a specific goal.

A most general execution sequence was defined by considering the above realizing that:

1. Decision making in the redirected program is to be a function of rigorous technical and economic evaluations which will use as input all of the R&D results available at any given time.
2. R&D results and economic projections in large multi-faceted projects are typically generated independently and the uncertainties associated with their accuracy can change significantly over the life of the project. An iterative decision making procedure that continues until sufficient confidence has been gained to permit reliable conclusions is required.
3. Design of thermal systems (including TES systems) requires an optimization procedure that balances the various thermal/hydraulic effects present.
4. Mathematical models exist, or could be generated easily for every element of the technical and economic evaluation procedure.

With these considerations in mind, an execution sequence was defined and is summarized in the flow diagram shown in Fig. 17. The sequence provides a flexible execution sequence that incorporates all of the generic steps associated with the development of TES media and/or systems R&D, as well as a framework for a consistent decision making

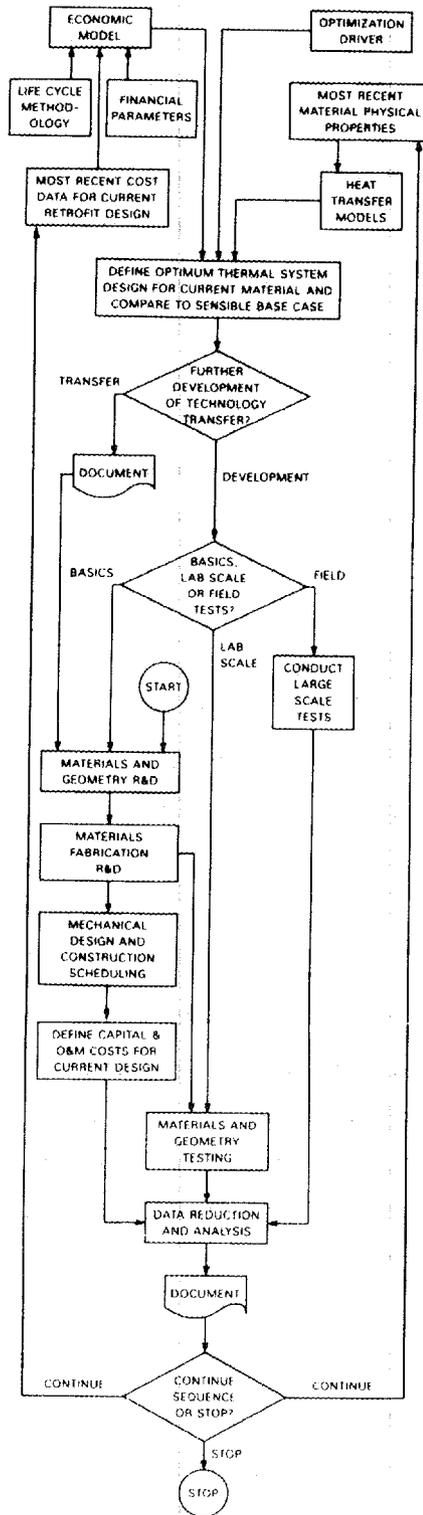


Fig. 17. Execution sequence for development of high temperature TES media.

methodology. As the figure shows, TES design optimization studies are the primary decision making tool regarding the viability of a particular latent heat storage media and/or system. These studies utilize information from each element of the project including economic and financial information, storage media thermophysical properties from lab scale research, and combined media/TES system performance characteristics from large scale field tests. In addition, the sequence allows for the iterative nature of R&D decision making by providing several opportunities during the development process where viability assessments can be made.

An examination of Fig. 17 reveals four basic project elements; optimization studies, thermal and economic models, storage media research and development, and large scale field testing of TES media and/or systems.

Optimization Studies: Economically based design optimization studies driven by the transient thermal response of a TES system provide the rigorous decision making tool required in the redirected project. Their objective is to compare life cycle costs between optimum sensible and optimum sensible/latent heat storage systems for an industrial application. There are two main justifications for conducting optimization studies:

1. Changes in bed geometry and/or system operating characteristics can effect life cycle costs as shown, for example, in Table 4.
2. There are many different mechanical designs and/or design constraints to be evaluated. These include the number and location of storage systems within the industrial plant, construction materials alternatives, the extent to which existing plant equipment can be used, and the number, type, and placement of latent heat storage media within the storage unit.

The design optimization procedure that will be used to accommodate all of these diverse influences is summarized in Fig. 18.

Thermal and Economic Models: The need for sophisticated thermal models also results from the requirement to implement a rigorous decision making procedure. Unlike traditional heat transfer equipment

Table 4. Thermal and hydraulic design considerations for thermal energy storage systems

Design change	Physical effect	Effect on life cycle costs
Increased Reynolds number	Increased heat transfer rate Increased pressure drop	Decreased fuel cost Increased pumping cost
Increased storage bed length	Increased total heat transfer Increased pressure drop Increased total mass	Decreased fuel cost Increased pumping cost Increased capital cost
Increased storage time	Increased total heat transfer Increased total viscous effects	Decreased fuel cost Increased pumping cost

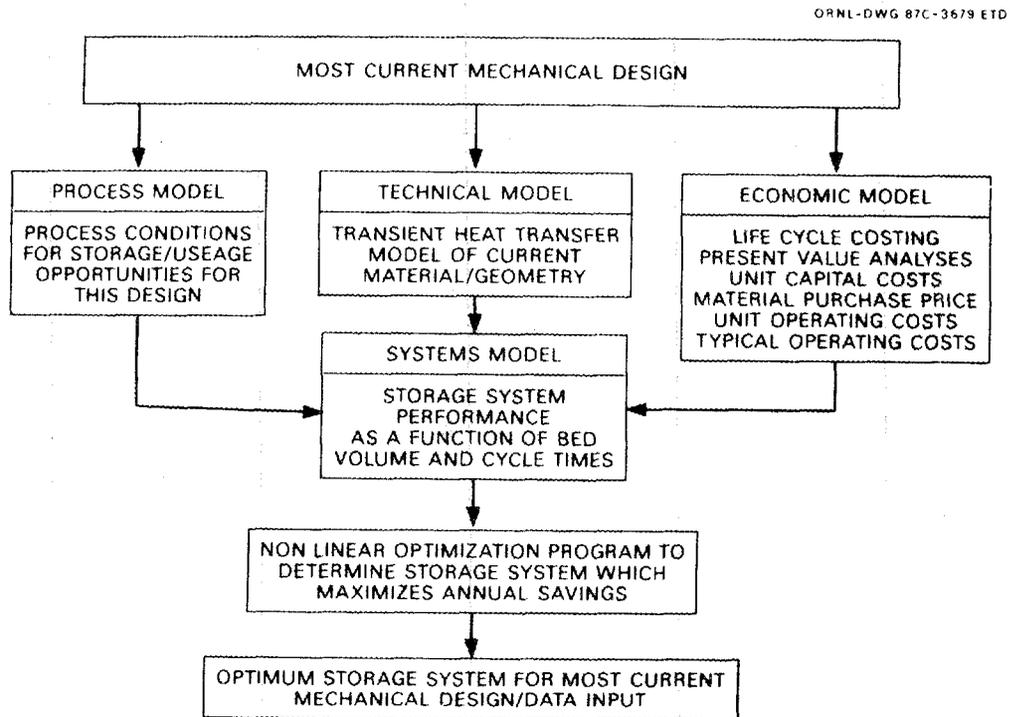


Fig. 18. Design optimization procedure for TES implementation in industrial application.

(i.e., condensers and cooling towers), thermal energy storage units have a constantly changing fluid/material temperature difference resulting in a constantly changing heat load. Since this characteristic cannot be predicted using steady-state correlations, transient models must be used. Transient thermal models also permit the operation of the storage unit to be defined in terms of geometry and process parameters; both of which may be optimization variables. System economic models are a further requirement. These models will be used to generate probabilistic cost estimates of components such as the storage media.

Large Scale Field Testing: Field testing as a key element in development of a high temperature TES system, exposes candidate storage media to realistic operating conditions and identifies systems considerations which cannot be evaluated in laboratory scale tests of small batches of storage media. These considerations include requirements for auxiliary components such as instrumentation and controls and realistic design and operating constraints such as (in the case of a brick plant) maximum combustion air preheat allowable.

Areas of responsibility were defined for all organizations currently associated with the Industrial TES Project. Each area, shown in Table 5, is in support of media development, the field test, or the evaluation procedure.

This sequence was implemented within the context of a specific goal: determination of the composition and geometry of the energy storage material that will be used in a large scale TES demonstration test scheduled for CY-89. This determination will be based on a comparison of life cycle costs between optimum sensible and optimum sensible/latent heat systems that could be installed as retrofits in an existing brick plant.

Table 5. Division of project responsibilities for the redirected high-temperature industrial TES project

Participant	Most general responsibility	Field test responsibilities
IGT	Materials research and development brick industry characterization	Metallurgical test requirements Acquire storage material for facility Contribute to facility RFP/ test plans Reduce test data
MSU	Thermal modeling field test support	Model storage unit and reduce data Design optimization of full size facility Contribute to facility RFP/test plans
ORNL	Program management thermal modeling optimization studies	Program management Write facility RFP/monitor construction Write test plans Coordinate data reduction/ analyses/documentation
PNL	TES market studies field test support	Define test environment/ instrumentation Supervise testing/data collection Contribute to facility RFP/test plans Market studies using reduced data
Schneider Brick & Tile	Industry consultant host plant for field test	Contribute to facility RFP/test plans monitor testing/data collection

Notes: IGT — Institute of Gas Technology
 MSU — Mississippi State University
 PNL — Pacific Northwest Laboratories

3. SUMMARY AND CONCLUSIONS

Progress to develop advanced TES technologies for the period of April 1986--March 1987 has been presented. Projects in the TES Program are directed to address specific issues that affect current TES performance and/or economics for low and high temperature systems in diurnal and industrial application areas.

Substantial progress was made to develop TES media and systems for high temperature industrial applications. Prior subcontracted efforts had examined, with limited success, the unique approach of quenching a molten AL/Si hypereutectic droplet such that the excess silicon would diffuse to the surface of the droplet and solidify, thereby forming an impermeable containment shell surrounding the AL/Si eutectic. During the reporting period, an in-house experimental project to determine the temperature conditions under which the silicon shell remained intact was completed. Through this study it was found that pellet temperature excursions up to 600°C would be acceptable during melting; mechanisms for pellet failure began to be seen at 650°C (thicker shells would be an option); above 700°C, the integrity of the shell would be quickly lost. Further development work on a unique TES media concept consisting of a salt or salt eutectic contained in the interstices of a sintered MgO pellet resulted in the identification of NaNO₃ as a promising salt for storage temperatures up to 400°C. Development work on the high temperature composite (Na₂CO₃/BaCO₃/MgO) has reached the field testing stage. Development of a plan for conducting this field test and integrating a series of activities directed at the central goal of the high temperature industrial subprogram was a major task completed during the reporting period. Continuation of the high temperature development activities/initiatives will proceed along the path defined by this plan.

In the low temperature regime, analytical procedures were developed to estimate heats of mixing in liquid-liquid systems. These procedures are based on the phase diagram (usually known) for the liquids and estimates of the component activity coefficients and will be used to determine the thermal storage capacity of liquid-liquid systems for potential use

in low temperature heating or cooling applications. A second significant development for low temperature applications was the identification of inert solvents for TES systems based solid/vapor sorption principles. As these solvents increase the adsorbent effective surface area, heat exchanger requirements are reduced to the point that successful development of a working sorption TES system may be possible.

In the reporting period, studies to determine the effectiveness of advanced heat transfer techniques coupled with TES systems were conducted. An experimental project to evaluate the heat transfer enhancement in pipe flow using a slurry showed substantial increases in Nusselt number over pure water; a somewhat smaller enhancement was seen through Stanton number correlations. Friction-reducing additives that reduce the slurry pressure drop in pipe flow was a major finding in this project. In a second effort, an assessment of state-of-the-art clathrate cooling system showed that despite efficiency improvements provided through the use of clathrates, significant cost and environmental issues remain unresolved. It is expected that clathrate system development will be hampered by pending production quotas on fully halogenated refrigerants.

In summary, TES is increasingly recognized as a technique for energy management, particularly in the area of cool storage for buildings. For these systems, electric utility incentives (high demand/energy charges and subsidies) are sufficient in some cases to make cool storage cost effective. Research has been performed and is continuing to develop advanced cool storage technologies to improve the performance and/or reduce the installed cost thereby widening the range of applications where cool storage is cost effective. In the industrial sector, the viability of TES systems for capture/reuse of waste heat is governed by the price of energy which is now used for heating in a particular industrial process. Since current energy costs are low, TES systems developed for this application must meet stringent cost and performance requirements. Simple PCM extrusions that can be used as packed bed TES systems continue to show the potential for meeting these requirements as

they are easily fabricated and expose large surface areas for high heat exchange effectiveness. Progress in the development of TES systems such as this will continue.

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