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High Temperature Thermal Properties

of Liquid Metals, Gases and Vapors

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Technical Scope and Research Objectives

(AT(11-1)-3027

The determination, interpretation and correlation of thermophysical properties and processes of fluids used or considered as nuclear reactor coolants and nuclear power cycle working fluids, with particular emphasis on high temperature and high pressure thermodynamic and transport properties of the alkali metals, most specially sodium, potassium, lithium and mercury.

U. S. Atomic Energy Commission support of this research program is gratefully acknowledged. The Principal Investigator has spent approximately 20% of his time during the 1972/73 academic year on this program, and 100% of two months during the summer of 1973.

ABSTRACT

During the 1972/73 contract year of AT(11-1)-3027, the main output was six reports presented and published at the Sixth Symposium on Thermophysical Properties (ASME, Atlanta, August 5-8, 1973):

- 1) COO-3027-85, "Corrected Thermal Conductivity of Li-
quids by the A.C.-Heated Wire Method"
- 2) COO-3027-4, "The High Temperature Vapor Pressure and the Critical Point of Potassium"
- 3) COO-3027-5, "The Vapor Pressure and Critical Point of Rubidum"
- 4) COO-3027-7, "Determination of Interfacial Tension at High Temperatures. Surface Tension of Potassium and Interfacial Tension of Li/LiCl to 850°C."
- 5) COO-3027-9, "Revision of High Temperature and Critical Properties of Cesium"
- 6) COO-3027-10, "High Temperature Saturated Phase Densities of Rubidium and the Critical Properties of Rubidium"

Two other reports were written for the Fifth International Conference on Heat Transfer (Tokyo, September, 1974):

- 7) COO-3027-13, "Interphase Phenomena and the Condensation Mechanism of Nitrogen Tetroxide Vapor"
- 8) COO-3027-14, "Heat Transfer in Pool Boiling and Condensing Selenium"

All of these papers derive from theses also being submitted as COO reports available for NTIS distribution for background details. The writing also involved substantial new experimental confirmatory work and correlating calculations.

In addition, programs on heat transfer by mercury vapor continued, and preliminary design studies and equipment assembly for the on-coming program on high temperature properties of sodium and lithium.

INTRODUCTION

Considerable progress has been achieved in the last year in the direction of reevaluation, final completion, and publication of the recent highest temperature results to date on physical properties and processes, particularly of alkali metals. In addition, other studies which were in the analysis and planning stage one year ago have been well commenced, and some should be completed in the 1973/74 year, particularly on sodium and mercury.

Analysis of the possibilities of extending to sodium and lithium the new methods developed in this laboratory for determination of vapor pressure and critical pressure and temperature clearly indicates that the critical point of sodium can be successfully reached with the methods and materials at hand, which should be of substantial value in LMFBF analyses.

In addition, the critical pressure of lithium will be approximated much more closely than heretofore, and possibly the critical point can be directly reached. This information should be of value in thermal designing for fusion reactor concepts employing lithium as coolant.

Finally, a modification of the recently developed methods of determining the vapor pressure and the saturated phase densities up to the critical point has been devised which it is believed can yield these same high temperature data previously obtained in the Liquid Metals Research Laboratory, and with more reliability than in the recent past, but can also yield the compressibility of the saturated and sub-cooled liquid phase and of the saturated and superheated vapor phase. The principal draw-back of the method is that with the same materials of construction (at present, chemically vapor deposited tungsten) the same high combinations of pressure and temperature cannot be reached, as the vessel's internal diameter should be larger than so far used, for good accuracy. However, presumably liquid phases at subcritical temperatures could be studied to above critical pressure and the vapor at supercritical temperatures and subcritical pressures, at least with Cs, Rb and probably K. It is being considered what alkali metals and ranges of study are most worthwhile, and what is the best equipment design for various sets of conditions.

Added details on these conditions are given next.

RESULTS AND PUBLICATIONS IN 1972-1973

A. High Temperature Thermodynamic Properties

1) Cesium. Earlier results on vapor pressure, including the critical pressure and critical temperature, as determined by Silver in 1967 (1,2) had required a 4-term equation in temperature T to give a good fit of his observed vapor pressure values, P . This type of equation yields an inflection point in a plot of $\ln P$ vs. $1/T$, rather than a straight or parabolic line. Other authors had not gone to as high a pressure, and had only employed 2- or 3-term equations, and evidently felt it inappropriate, on theoretical grounds, to utilize a 4-term equation. Since Silver's work, our experimental equipment and procedures have improved considerably.

Accordingly, a verification study, duplicating Silver's range of work, was carried out by Das Gupta (3,4) with an improved furnace, pressure tube and injector. Unfortunately, an experimental problem was encountered in that the pressure tube tip thermocouples above about 2850°F. all began to read too high or too low and not in any agreement. However, whenever the tip temperature was decreased below about 2800°F., they all read reasonably and in close agreement. It was concluded that the one alumina thermocouple sleeve inadvertently left at the end of one of the thermocouples during installation had melted and fluxed with some of the other (magnesia) tubes, forming a compound with a melting point in the range of 2800-2850°F., which was shorting or grounding out the thermocouple wires when it was molten. Thus, no vapor pressure points above 2850°F. could be obtained with this installation. It also proved impossible to chip or melt and drain off the compound, present only as a thin film on the tungsten tube tip. However, good vapor pressure points below 2800°F. were readily obtained. Also, the critical pressure could be observed with full accuracy without needing the tip temperature. Since these results very closely duplicated those reported earlier by Silver, it was adjudged not necessary to again repeat the Cs vapor pressure tests on a third pressure tube, but rather to preserve the two remaining tubes for Na and Li. Thus, the former vapor pressure equation and critical pressure and temperature of Silver (2) were allowed to stand. However, by combining all data into one pool, slightly altered results were obtained; although there was no increase in the number of vapor pressure points above 2850°F., therefore some added weighting of the lower pressure points, the new equation would have to be considered as based on more points, therefore more reliable than the previous one. The

pooled results for Cs now give (4) for P in psia and T in °R:

$$\ln P_V = 1.6489 - 12322/T + 1.12 \ln T - 2.9044 \times 10^{-64} T^{17.5} \quad (1)$$

$$P_c = 1701.3 \text{ psia} \quad (2)$$

These P_V results were analyzed by calculation of the heat of vaporization of Cs monomer at 0°R, showing excellent consistency by this thermodynamic procedure (4).

Results on the saturated liquid and vapor phase densities of Cs up to the critical point had also been reported from this laboratory by Oster by the tilting capsule method (5,6). Unfortunately, in his pioneer effort, he was only able to get one run, which yielded one equation in two unknowns, the saturated liquid and vapor densities, at each temperature. To obtain the separate phase densities, it was necessary to make some additional assumption as to either or both densities in the (high temperature) range of these tests. At the time, either a rectilinear diameters relation derived from prior results in this laboratory by Hochman (7,8) was employed (5) or one based on a later study and extrapolation by Ewing, Stone et al (9) was utilized (6). However, recently it was decided that to make most accurate use of Oster's results it is preferable to employ for the other information the forms of well-established correlating relations for Cs vapor and liquid. Accordingly, the following relation was used for both liquid and vapor:

$$\rho = \rho_c + \alpha(1-T_R) + \beta(1-T_R)^{\gamma} + \epsilon(1-T_R)^2 \quad (3)$$

which is of fairly general acceptance for alkali metals(10). The procedure consisted of assuming a set of coefficients for eq. 3 for the vapor and another for the liquid and then calculating the vapor and liquid densities, and from them the tilt angle by the moment theory (6). The standard deviation between these calculated and the observed tilt angles was then computed. The process was repeated with other values of the coefficients in eq. 3, which include ρ_c , T_c , and α , β , γ and ϵ for the vapor and for the liquid, namely ten coefficients. In each case the constraint that the curve of liquid density, ρ_L , must pass through the single directly-observed value (when the capsule just reach horizontality) of 0.4853 g/cc at 3225°F. is maintained. The lengthy calculation ends when the minimum σ has been found between the tilt angles, and yields an equation of state for each saturated phase. Although a moderate inversion appears in P_V at about 2500°F., this procedure is believed to yield the most reliable high temperature saturated density results feasible from the Cs tests so far in this laboratory.

2) Rubidium. The dissertation by Chung (11), which yielded the high temperature and critical density of the saturated liquid and vapor phases by our tilting capsule method (5,6), had been completed last year. In this year a briefer version was prepared and published (12). It is noted that the critical temperature was reliably obtained, by two separate runs in excellent agreement, as 3331°F., and subcritical ρ given.

The thesis of Bhise (13) had presented the first vapor pressure data that reached the critical point of Rb. However, due to an inadequate understanding at that time of the proper procedures for the new "pressure tube" method that was developed, oil penetrated down towards the tip of the tube through freezing cracks when, between runs, the Rb was allowed to freeze towards the tip instead of away from it. Thus, on a later high temperature run the oil carbonized and the tube became plugged, which cut short the experimental program somewhat. A final result of the thesis was close agreement between the two supercritical points obtained, that the critical pressure was 1943 psia.

However, there was a possible discrepancy in that the lower critical pressure point had a reported tip temperature of 3769°R, which would have been expected to be above the critical temperature. Whereas, the highest subcritical point at 1853 psia had a tip temperature of 3732°R. Plotting the subcritical points (13) as $\ln P$ vs. $1/T$ (see Fig. 1) clearly showed that a smooth curve through them, accepting the 3732°R point, would hit P_c above 3769°F. Furthermore, the independent study of density by Chung (11,12) gave 3791 as T_c , which plots smoothly with the subcritical points, and clearly concluded that the 3769°R observation is the temperature reading in error, and that T_c stands at 3791°R. However, when the opportunity comes up in scheduling, several more check runs in the critical region of Rb will be carried out, as they were for K. In the meantime, the critical point and the vapor pressure curve from about 1800°F. up may be considered as quite well established, as detailed in the third report prepared for the Atlanta symposium (14).

3) Potassium. Since potassium has been seriously considered and evaluated, both as a nuclear reactor coolant (ORNL) and a turbine working fluid (GE), it is perhaps fortunate that it was not studied until after Cs and Rb. The reason was that its critical pressure and temperature were expected (and found out) to be higher. With this timing, a material of construction, chemically vapor-deposited (CVD) tungsten, became available for

the "pressure tube," which eliminated that main materials problem at about midcourse in the K program. In addition, the prior experience with Cs, Rb and K at that point taught how to avoid plugging of the pressure tube, so that in the latter part of the K program there has been more rapid and reliable progress.

The first vapor pressure and critical point study (15) was completed last year by Jerez, and provided the backbone of the K results. Subsequently, Freyland and Hensel (16) published work claiming a critical point for K about 150°F. and 70 psi lower than our values. Since this discrepancy could only be effectively resolved by data very close or at critical point conditions, a new series of runs was carried out in that range. A total of ten distinct points was obtained at temperatures higher than Freyland and Hensel's indicated t_c , yet below our well established P_c . The high concentration of our near-critical points also permitted very precise extrapolation of the vapor pressures to P_c to yield t_c . These final values for potassium are reported (17), in excellent agreement with (15), as:

$$\begin{aligned} P_c &= 2378 \pm 4 \text{ psia} \\ t_c &= 3646 \pm 5 \text{ }^\circ\text{F.} \end{aligned}$$

Vapor pressure equations and interpolation tables were included.

A study of the surface tension of K by Chung (18) has been carried out as a standardization study of a new design of interfacial tension apparatus suitable for liquid metals and fused salts, which employs the maximum bubble pressure method. However, by comparison with other published surface tension results for K, the values here determined had significance in themselves, in that they agreed rather closely with some other work, with additional published surface tension values for K being considerably higher or substantially lower. Erroneous surface tension results will generally be too low, as σ -depressing impurities tend thermodynamically to concentrate at the surface and σ -raising impurities to flee the surface and have little effect.

The present and the literature results were restudied as the above study was prepared for publication, with the following conclusions (19):

In the case of K, published results by Solov'yev and Makarova, and others by J. W. Cooke, both fitted to straight lines, agreed well with the present slightly curved results over at least parts of their temperature ranges. All other results disagreed with these and among themselves, so that the lower ones can presumably be discarded due to surface-concentrating impurities, and the higher ones due to gross impurities or to apparatus

malfunction. The correlating equation, for 100 to 850°C., is

$$\sigma_K = 126.1 - 0.1036 t + 0.000040 t^2$$

in dynes/cm. for t in °C.

Interfacial tension data for the system Li/LiCl were also obtained for use in designing a high energy, rechargeable primary battery employing Li and Cl₂. The concept of a double-capillary interfacial tension cell to avoid measurement of the capillary tip submergence was well demonstrated, and seems suitable for future surface and interfacial tension measurements at high temperatures.

4) Thermal Conductivity by A.C. The sixth and final paper presented for the Atlanta Thermophysical Properties Symposium presented new, corrected experimental results for the thermal conductivity of electrically non-conducting liquids by the A.C.-hot wire method developed in this laboratory. In an attempt to verify whether this method, previously developed to decrease radiation correction for high temperature data, would also be better with liquids than the conventional D.C.-heated wire, tests on a group of organic thermal transport liquids had been carried out (20). It later developed that the Dowtherm A values agreed poorly with the consensus of literature results. To confirm or correct the prior results on the organic liquids, some were repeated by Siegel (21). The new values, on Dowtherm A and on Isopar M, are much more credible, and it is suspected that some undetected program error was present in the complex computer program for data reduction previously used, and is now corrected. Though the method is meritorious for gases at high temperatures subject to thermal radiation, it seems that the less complex D.C. method will be better under other conditions.

B. Other Completed Research

Additional studies under this contract that were completed as dissertations in the contract year 1971/72 were rewritten, with revisions, for publication in 1972/73, thus constituting part of this year's overall effort. Both of these papers were accepted at the abstract level, and have been completed as mats for final review for the Fourth International Conference on Heat Transfer (Tokyo, September, 1974).

1) Dynamics of Condensation of Metal Vapors. Initial results on condensing heat transfer of alkali metal and mercury vapors with Misra (1953-1956) and Englebrecht (1958-1961), subsequently also observed by other authors, indicated, after making the "temperature jump" correction, accommodation and condensation coefficients of the vapor molecules on impinging with the metal condensing walls that were consistently at or near unity

at low condensing pressures (1 to 10 mm Hg range). However, as the pressure was raised towards atmospheric, the above calculated coefficients dropped towards 0.1 and lower. Since the principal molecular difference in the vapor over this pressure range is that it varies from a negligible concentration, of polymer molecules to only an appreciable concentration, it was early planned to carry out an analytical study of available condensing metal heat transfer data from this standpoint.

Quantitative experimental verification of any such theory with metal vapors would seem unlikely due to difficult experimental conditions and low polymer contents, so the concept of modeling the phenomenon with condensing $\text{NO}_2\text{-N}_2\text{O}_4$ emerged. A full study has now been completed (22) and a concise version prepared for publication (23).

In the theoretical analysis, a complete solution was obtained by applying Boltzmann's integro-differential equation to the vapor, subject to the boundary conditions at the vapor-liquid interface implied by the condensation and the energy-accommodation coefficients. The 13-moment expansion by Grad was utilized. The result is complex, but is apparently the most accurate to date for interphase transport with a gas.

In the experimental phase, the temperature profile in the $\text{NO}_2\text{-N}_2\text{O}_4$ vapor was measured by a microprobe in the last millimeter of travel before condensation. The vapor was found to be subcooled near the interface. By comparison with theory, the result was obtained that the condensation coefficient of the monomer specie is several times larger than for the dimer, surprising since the liquid is almost pure dimer. However, this conclusion agrees with all observed effects.

2) Two-Phase Heat Transfer Properties of Selenium. Selenium has been suggested as a possible thermodynamic working fluid for high temperature space nuclear power cycles, similarly to sulfur but in the boiling range of Cs and Rb. Actually, containment problems seem to make it impractical for now. However, it has a complex molecular structure of long polymer chains and polymer rings, in both the vapor and liquid phases, and may yield a useful contribution in interpreting two-phase heat transfer of metals, as mentioned under B-1 above. For this possible contribution to theory, as well as to have the experimental results for possible thermal design, a program was carried out (24) and condensed for publication (25).

Stainless steel 316 was found to be the most resistant heat transfer surface, even including a number of refractory alloys. Though corrosion was at the rate of several mm per year, boiling and condensing heat transfer coefficients were not affected significantly. The coefficients themselves were in the range of 100 BTU/hr x ft² x °F at 10 mm Hg pressure, increasing to as much as 1000 at atmospheric pressures. Though low, they would improve further at power cycle pressures if corrosion can be controlled. The results were interpreted qualitatively in terms of the chains and rings structure.

C. Mercury Vapor Program

A program is also under way in which previous studies of mercury vapor up to atmospheric pressure are being pushed to the range of 500 psi and higher.

1) Thermal Conductivity. A recent study (26) employing the A.C.-heated wire method obtained the thermal conductivity of Hg vapor. It established experimentally the decrease obtained with increasing superheat at constant pressure due to decreasing dimer content of the vapor. However, the result agrees more with a "frozen vapor" monatomic model than with simple theory. Also, at atmospheric pressure, the dimer content of the vapor is only about 1%, and the experimental increase due to dimer is low and not sufficient for desirable accuracy in interpreting the molecular effect of polymerization. In addition, any future applications of mercury as a coolant or for topping cycles, may well employ much higher pressures, as the critical pressure is some 22,000 psi. Accordingly, a program of measuring the thermal conductivity of mercury vapor to much higher pressures than atmospheric is under way, with an objective of at least 500 to 1000 psi, and as much higher as possible. A first run was carried out before the student, J. R. Siegel, reported for a period of active military service. Resumption of the program is expected shortly.

2) Condensation of Mercury Vapor. As discussed under B-1 and C-1 above, condensing heat transfer data for mercury at fairly high pressures should be helpful in extending analytical studies of the molecular mechanics of condensation of metal vapors, and in providing empirical information for the thermal design of mercury vapor condensers. Accordingly, an experimental study of mercury vapor condensation at up to 500 psia is under way by S. C. Hsieh.

Previous SNAP experience by NASA and GE has indicated that clean tantalum is an ideal surface with mercury, yielding good wetting, complete corrosion resistance, and high thermal conductivity. A tantalum tube with smooth finish and high dimensional accuracy, 12 inches long, 0.625 inches O.D. and 0.040 inches in wall thickness, has been procured for the condensing surface, and nominal 3 inch Schedule 80 No. 1316 Stainless Steel, welding nipples, caps, tee and 45° elbow for the body of the mercury container. The design follows the conservative ASME Code for long-time usage at 500 psi and 1200°F.

Hg will boil in one (the lower) end of the apparatus, with electric heating through the wall. The condensing tube, air cooled internally, will be located across the other end, and can be operated horizontal, vertical or in between by appropriately turning the apparatus around a 45° axis. The whole apparatus will be in a vacuum box in a well-ventilated room for control of possible Hg leakage.

Instrumentation has been carefully planned in light of the previous condensation experience of this laboratory (22-25, etc.), and will include wall temperature measurement by direct and differential thermocouples, and actual superheated as well as saturation temperatures of the Hg vapor.

D. Sodium and Lithium Program

During 1972/73 considerable planning and preliminary design and procurement and some fabrication and testing took place towards high temperature Na and Li property determinations. In particular, a 3000°C rating tungsten mesh furnace and controller has been acquired, a reconditioned-to-new Barnes radiation pyrometer, a 5000 psi self-balancing Dresser "Digigauge" accurate to about 1 psi (0.02% of full scale) and two CVD tungsten "pressure tubes," 1/16 in. ID x 0.32 in. OD x 15 in. long. In addition, several other high-range radiation pressure gauges on Government surplus have been requested for back-up and broader range use. For instance, whereas in the past we concentrated on simple, and therefore more reliable, equipment, aimed at our prime objectives (such as the critical region of an alkali metal), now we will record data also in other regions, such as vapor pressures down to atmospheric. This will not only provide new data of good precision in these other regions, but serve for intercomparison of results, to facilitate evaluation of absolute accuracies. The principal difficulty that has been recognized to date, and is being

attacked, is that of assuring reliable radiation pyrometer operation and accuracy, since tests will be getting beyond the temperature range of W-Re thermocouples, so satisfactorily relied on over the last few years.

Charles F. Bonilla
Principal Investigator

Figure 1

Vapor Pressure Results on Rubidium (14), Showing Curve Passing Smoothly between Highest Subcritical Point at 3732°R, and Critical Temperature at 3790.7°R (11,12).

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