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Pressure Phase Lines and Enthalpies for the α - β and β -Liquid Transitions in Beryllium

Albert Abey

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Pressure Phase Lines and Enthalpies for the α - β and β -Liquid Transitions in Beryllium

Abstract

The effect of hydrostatic pressure on the α - β and β -liquid transition temperatures in Be was measured in a gas pressure system. Differential thermal analysis was used in the pressure range from 0.1 MPa to 0.7 GPa. For the α - β transition, $dT/dP = 43 \pm 7$ K/GPa; for the β -liquid transition, $dT/dP = 35 \pm 7$ K/GPa. Although it is possible that large systematic errors may arise from experimental procedures, our results are seriously at odds with those of other investigators. Transition enthalpies for the α - β and β -liquid transitions were 1.9 ± 0.2 and 2.2 ± 0.2 Kcal/g·m, respectively, at a pressure of 0.1 MPa.

Introduction

Our investigation was initiated to obtain an increased understanding of the effects of pressure on the behavior of beryllium in the temperature range near its melting point. In particular, our goals were to investigate the effect of pressure on the α - β (solid-to-solid) and the β -liquid (solid-to-liquid) transition temperatures. Our study also included a determination of the α - β and β -liquid transition enthalpies at a low pressure, 0.1 MPa.

Although such measurements have been previously reported by other investigators, the pressure behavior of beryllium near its melting point is still very much in question. Beryllium has the reputation of being a difficult material with which to work. For our investigations of the high-temperature phase diagram of beryllium under varying static-pressure conditions, this reputation is easily upheld.

Francois and Contre¹ studied the α - β and β -liquid transition temperatures in beryllium as a function of pressure in a solid-medium pressure system. The phase transitions were detected by changes in electrical resistance in a beryllium wire that was heated through the transition temperatures while under pressure. These researchers observed the β -to-liquid transition at 1558 ± 100 K

and reported a positive pressure slope of approximately 40 ± 20 K/GPa. The α -to- β transition temperature was shown to have a slope of -40 ± 20 K/GPa. However, the phase lines reported by Francois and Contre in their preliminary report (no final paper was published) contained large error bars and numerous disclaimers. In addition to the obvious problems associated with measuring the pressure phase lines on two transitions that are variously reported to be separated by only 10 to 25 K at 1550 K, the experimental approach and techniques used by these researchers place their results in serious question.

A further indication of possible problems with published results in this temperature range arises from the enthalpies measured by Loasby and Dearden.² Their outcomes differ, by a factor of 3, from the total enthalpy for the transitions reported by Kantor et al.³ Loasby and Dearden determined the transition enthalpies using differential thermal analysis in an intermediate vacuum, whereas Kantor et al. measured the combined enthalpies in a drop-calorimetry experiment. Our investigation was an attempt to resolve the discrepancies in these previously published reports.

Experimental Approach

After considering several experimental approaches, we used differential thermal analysis (DTA) in a hydrostatic-gas-pressure system. Although there are problems in using a gas system, as there are in any pressure system, we believed that more reliable data could be obtained over the limited pressure range possible in a hydrostatic system. Both DTA and high-pressure gas technology have been used successfully in the past by other researchers and at LLNL. However, combining the two technologies in a manner that yields unambiguous results has proven to be difficult.

Apparatus

The sample-furnace assembly is shown in Fig. 1. From published reports, we assumed that at high temperatures Be would react chemically with most substances except for BeO and the noble gases. On the basis of this assumption, a 0.2-g cylindrical sample of Be (7.4-mm length, 4.2-mm diam) was enclosed in a BeO furnace tube (4.5-mm inner diam, 6.4-mm outer diam). The sample was enclosed with BeO end caps. Both end caps and sample contained a 0.84-mm-diam hole along

Figure 1. Sample-furnace assembly.

their axes. A BeO tube with an inside diameter of 0.3 mm was placed through the hole. The sample thermocouple of Pt-Pt-10%Rh was passed through this tube by entering one leg from each end. Thus, the junction of the sample thermocouple was positioned, as closely as possible, in the middle of sample and halfway between the end caps. Alumina cylinders were placed against the BeO end caps in the BeO furnace tube.

In addition to the sample thermocouple that passed through the alumina cylinders, a Pt-Pt-10%Rh thermocouple was placed in each alumina cylinder. These were located approximately 5 mm from the BeO end caps. One of these thermocouples served as the reference thermocouple in the DTA setup, and the other was used as input to the temperature controller.

The thermocouple leads were continuous except for a Pt-Pt or a Pt-10%Rh-Pt-10%Rh connection from the sample assembly and pressure plug to the ice-bath cold junction. The resistance to ground for each thermocouple was $>10\text{ M}\Omega$. The effective shunt resistances between the thermocouple legs (through the sample holder, for exam-

ple) are not known at conditions of high pressure and high temperature.

To minimize temperature gradients in the sample region, the number of furnace windings around the 6.4-mm-diam BeO furnace tube varied with distance from the ends. Approximately twice as many windings per mm were located on the ends of the tube than in the middle section. More complex and, perhaps, effective schemes of controlling the temperature gradients were not attempted due to the limited number of available electrical leads. In addition, both input-power levels and temperature gradients along the sample holder are a strong function of pressure. The net effect of these relationships on experimental results are uncertain (see Results section).

The assembly was placed in a Zr_2O_3 cylinder. All additional volume in and around the assembly was filled with insulating material to minimize heat losses and temperature gradients.

A schematic of the standard 1-GPa high-pressure system used in the experiment is shown in Fig. 2. The vessel was externally water cooled and had an inside working diameter of 38 mm.

Figure 2. Schematic of the high-pressure gas system.

The maximum pressure of 0.7 GPa used in this study was measured with a Bordon tube gauge.

Temperature was controlled along the desired path by a programmable temperature controller. The output of the temperature controller regulated the power to the furnace through a silicon controlled rectifier pack. Temperature deviations from the desired temperature path were <0.1 K at the position of the control thermocouple.

A schematic of the electronics is shown in Fig. 3. The thermocouples were connected in the standard DTA manner to two digital voltmeters. The analogue outputs of the digital voltmeters (one was the sample temperature and one was the temperature difference between the sample and reference thermocouples) were fed to several strip-chart recorders that displayed the data by means of different scales.

Procedure

After the sample-furnace assembly was placed in the pressure vessel, the system was purged several times with low-pressure gas. The purge gas was the same as the pressure medium, generally argon. For each data run the pressure was set, and temperature increased along a linear path. The rate of change for increasing temperatures was usually 4 K/min in a range that extended from about 50 K below the transition region to 50 K above that region. During the temperature increase, the pressure changed slightly, either from heating or from a leak. Such changes were small, and the pressure was recorded at the beginning of the first transition. The cooling rate was generally 20 K/min. This cooling rate not only saved time but also yielded useful additional information. Samples were always

Figure 3. Schematic of the electronics. The curves on the strip-chart recorder shown at bottom left are the sample-reference temperature difference and the sample temperature. The curve on the recorder shown at bottom center is the sample temperature on an expanded scale.

taken through the transitions at least once before data were recorded.

Two typical DTA traces are shown in Fig. 4. The trace in Fig. 4(a) was obtained at a pressure of 0.1 MPa; that in Fig. 4(b) was obtained at a pressure of 0.2 GPa. At 0.1 MPa the two transition temperatures are completely separated and unambiguous. This was true for the transitions during both increasing and decreasing temperatures. At 0.2 GPa the transitions are no longer completely separated, indicating a possible increased temperature gradient across the sample. The transitions observed during cooling were, however, still clearly separated.

An interesting and useful observation with respect to the transitions that occur during cooling arises from these and all other traces obtained during the study. With cooling, both phases are metastable to a degree, and temperatures at which

the transitions occur are not directly related to the thermodynamic transition temperatures except that they are, of course, lower. The interesting point is that the maximum temperature reached after the sample undergoes a metastable cooling transition is the same, within narrow limits, as the transition temperature observed during heating. Our reasoning is that when the sample undergoes a rapid cooling transition at a temperature below the true transition temperature, heat is evolved in a very short time. This energy increases the sample temperature up to the thermodynamic transition temperature. The sample temperature cannot increase above the thermodynamic transition temperature because there is insufficient energy available to take the sample through the transition.

This observation is important because the heating transitions became increasingly less distinct as the pressure increased, while the peak

Figure 4. Typical DTA traces. (a) Trace of the sample-reference temperature difference (top) and of the Be sample temperature (bottom) taken at a pressure of 0.1 MPa. (b) Traces taken at a pressure of 0.2 GPa.

temperatures during cooling transitions remained separable at all pressures. The correlations between heating and cooling transition temperatures held for all cases and at all pressures. An expanded temperature trace for the sample shown in Fig. 5 demonstrates this correlation for a typical low-pressure data run.

Figure 5. Expanded temperature trace for the sample obtained at a pressure of 0.1 MPa shows good correlation between heating and cooling transition temperatures.

Results

Determination of Transition Temperatures

Useful results were obtained from about 40 experimental runs. Most tests were carried out with increasing pressures; however, some used decreasing pressures, and in combined runs increasing pressure was followed by decreasing pressure or vice versa. Failures in the sample assembly severely limited the number of data runs on a given sample. All runs generated data that were qualitatively the same: that is, for any given experimental run the general characteristics were the same for both the α - β and β -liquid transitions and for data obtained from either heating or cooling.

Data from the same three runs for increasing pressures are shown in Fig. 6. Figure 6(a) shows the α - β (solid-to-solid) transition; Fig. 6(b) shows the β -liquid transition. In general, transition temperatures first dropped with increasing pressure,

up to a pressure between 0.05 and 0.1 GPa, and then transition temperatures increased with increasing pressure up to the maximum experimental pressure. The pressure at which the transition temperature started to increase varied between experimental runs. However, in a few runs, including one in Fig. 6, the transition temperatures increased with pressure over the complete pressure range.

Figure 7 shows the α - β transition temperatures for pressures first increasing to 0.2 GPa and then decreasing to 0.1 MPa. Here, the difference between transition temperatures for increasing vs decreasing pressure is 3 K. In general, this difference occurred even though the sample was taken through the transitions at 0.1 MPa at least once before data runs were started.

Temperature gradients in the sample region were generally a strong function of pressure, especially between 0.1 MPa and 0.1 GPa. The problem of temperature gradients persisted over all data

Figure 6. Data from three experimental runs for the (a) α - β (solid-to-solid) and the (b) β -liquid transition temperatures. Transition temperatures are plotted vs pressure.

runs and through all minor modifications of the sample assembly. (The use of BeO in the assembly made major modifications a long-term task.) In addition to the problem of temperature gradients, discussed in more detail below, the sample moved slightly in the sample holder each time it underwent a phase transition. It is likely that both of these effects influenced the data, especially at lower pressures.

Figure 8 is a schematic representation of the temperature gradient in the region of the sample. The solid line indicates a possible temperature profile. This profile is a function of both pressure and temperature, although the temperature dependence is small in the temperature range near the melting point of beryllium.

During heating, the phase transition generally starts at the warmest part of the sample and propagates along the sample as the temperature increases due to the global temperature increase dictated by the temperature controller. At the same time, a temperature wave is propagated down the temperature gradient in advance of the phase transition. In the case of position 1 in Fig. 8, the sample thermocouple indicates a temperature very near the transition temperature since the temperature gradient is shallow. If the relative sample-gradient relationship is that indicated by position 2, then the phase transition starts at the warmer end, and a temperature wave will travel from the warmer end to the cooler end. In this

Figure 7. Transition temperature for the α - β transition varied by 3 K for pressures first increasing to 0.1 GPa and then decreasing to 0.1 MPa.

case the sample thermocouple will indicate the phase transition at a temperature below the actual transition temperature. (The temperature wave travels through the sample faster than the phase transition.)

Figure 8. Temperature gradient in the region of the sample as a function of the distance along the furnace axis.

If the temperature gradient were identical at all pressures, there would be an offset in the transition temperature, but the variation with pressure would not be affected. Unfortunately, the temperature gradient during the experiment varied from run to run in a manner that was somewhat unpredictable. Although the variation in the temperature gradient was relatively small, it could not be determined accurately due to experimental limitations. In effect, the variation in the temperature gradient with pressure makes it impossible to precisely determine changes in the transition temperature at low pressures.

While our uncertainty with respect to the low-pressure data is large and unpredictable, the low-pressure data may be an anomaly caused by changing temperature gradients. If so, then both transition temperatures would increase with pressure even at low pressures. In the discussion that follows, we assume this to be the case. The values for the slopes were taken from that region of the curves for transition temperature vs pressure where the slope is positive and relatively constant.

Slopes for the α - β and β -liquid transitions for both increasing and decreasing temperatures are shown in Table 1. The slopes for increasing temperature are slightly but consistently larger than those for cooling for both transitions. (The discrepancy is probably a result of different temperature gradients that arise during increasing and decreasing temperatures.) The errors are the variations from run to run and do not include systematic experimental errors. The slopes predict that the α - β phase transition will disappear in the neighborhood of 2.5 GPa.

Our slopes for the phase lines are seriously at odds with the experimental results of other inves-

tigators. Figure 9 shows the published phase lines of Francois and Contre¹ together with those from the present study. The phase lines for the β -liquid transition are in reasonable agreement. The Francois and Contre phase line for the α - β transition has a negative slope and suggests the possibility of an α - β phase transition at room temperature at a pressure around 20 GPa. Our results indicate a positive slope with the α - β phase disappearing near 2.5 GPa.

Two x-ray investigations of beryllium at temperatures increasing through the α - β phase transition have been reported.^{4,5} In both of these studies, a decrease in volume was observed during the HCP-to-BCC (α - β) transition. The negative volume change is consistent with the results of Francois and Contre and is unreconcilably inconsistent with the results of our investigation. The situation is further complicated by three recent sets of experimental data.

Figure 9. Transition temperatures vs pressure. The dashed lines are the results of Francois and Contre.¹ The solid and dotted lines are the results from the present study.

Table 1. Slopes for the α - β and β -liquid transition temperatures.

Transition	Temperature change	Slope (dT/dP) (K/GPa)
α - β	Increasing	45 ± 7
α - β	Decreasing	42 ± 5
β -liquid	Increasing	36 ± 5
β -liquid	Decreasing	34 ± 7
$T_{\beta \text{ liquid}} - T_{\alpha \beta} \cong 20 \text{ K}$		
$P(4.5 - 3.6) \cong 20 \text{ K}$		
$P \cong 2.5 \text{ GPa}$		

Reichlin⁶ monitored the room-temperature electrical resistance of beryllium in a diamond-cell apparatus to pressures above 40 GPa. Using a 4-wire resistance technique, only a slight continuous decrease in resistance with increasing pressure was observed over the pressure range. Ming and Manghnani⁷ used x-ray diffraction to determine the structure of beryllium at pressures up to 28 GPa at room temperature in a diamond-cell apparatus. They observed a phase transition from HCP to a distorted HCP at pressures between 8.6 and 14.5 GPa. This transition has a small volume change (<0.5%) and was the only transition detected over this pressure range. Wise et al.⁸ conducted a series of shock experiments and found no evidence of a phase transition in the pressure range from 6 to 35 GPa.

From the experimental evidence to date, no clear-cut picture seems to emerge that can explain the pressure behavior in the temperature range near the melting point of beryllium. The volume changes calculated from the Clausius-Clapeyron equation for the two phase transitions from the experimental results obtained in our work are

$$(\nabla V/V)_{\alpha-\beta} = 0.048$$

and

$$(\nabla V/V)_{\beta-1} = 0.046 .$$

The calculated magnitude of the volume change associated with the α - β transition agrees reasonably with the value found by Ref. 4, but, of course, the sign is different. It is possible that impurities could affect the α - β phase line in some manner not yet understood. Several beryllium samples were quantitatively analyzed for impurities using x-ray techniques. A typical analysis is shown in Table 2 for both the outside surface and the inside of a sample used in our study. Results for the inside were obtained from a surface exposed by cutting the beryllium sample in two. Also included is an analysis for an unused beryllium sample. No unexpected impurities appear in the results.

Determination of Enthalpies

The enthalpies for the α - β and β -liquid transitions were determined at a low system pressure and in the same apparatus that was used for the determinations of phase lines. Enthalpy data were obtained by comparing the areas under the DTA

Table 2. X-ray analysis of impurities for a beryllium sample used in the phase-line determination and for an unused beryllium sample.

Element ^a	Experimental Be sample		Unused Be sample (ppmw)
	Outside surface (ppmw)	Inside surface (ppmw)	
Si	150	30	60
Fe	60	<60	<60
Ni	30	15	30
Ti	<30	<30	<30
B	8	<8	8
Ca	<8	<8	<8
Cu	8	8	8
Mo	8	<8	30
Ag	6	6	8
Mg	6	8	3

^a The following elements were not detected: Na < 800 ppmw; Nb, Sb, and Zr < 80 ppmw; Pt, Al, Ba, Cd, Pb, and Zn < 8 ppmw; Mn and Sr < 3 ppmw.

curves for Be with the area under the DTA curve for melting Ag. Since only one sample at a time could be placed in the apparatus, the Be and Ag samples were not run together during the same experiment. Such separate determinations probably did not contribute a significant source of error. Several runs were performed on both Be and Ag, and the results from the various runs were consistent within narrow error limits. The consistency is due, at least in part, to the small temperature gradients within the sample holder at the low Ar pressure during these tests.

The Be and Ag samples used in the enthalpy determinations were treated in the same manner. Samples were loaded into the sample holder in the normal way. The vessel was purged several times with low-pressure Ar to remove as many impurities as possible. After the Ar purge, the vessel was closed with the pressure at 0.1 kPa. While sealing the vessel allowed the pressure to increase only slightly as the sample temperature was taken through the transition temperatures, the sealing limited impurities that might contaminate the sample to those remaining in the system. After purging and sealing the vessel, the sample was taken through the transition temperature once before enthalpy data were taken.

Enthalpy data were obtained by recording the DTA trace as the sample was taken through the transition temperatures 10 times. The first two data runs were taken at a rate of 4 K/min, and the remaining eight were obtained at 2 K/min. At

these rates of temperature change, the DTA peaks for the two Be transitions were well separated, and no error arising from an overlap of transitions entered the calculations. For the Ag standard, the DTA peaks were very reproducible over the 10 runs. The peaks for the Be showed a monotonic decrease in amplitude as the runs continued. The decrease is attributed to Be loss during the runs either from sublimation, oxidation, or both. The amplitudes used for the Be transitions were obtained by extrapolating to the first sample-seating run.

Results obtained for the Be runs at low Ar pressure in the enthalpy determination were in good agreement with the large number of low-pressure runs from the phase-line experiments. The two Ag standard samples were used in the enthalpy determinations and yielded similar results.

Several special runs were carried out after the ten enthalpy runs. The peaks shown in Fig. 10 for one of these runs are typical of those obtained from the enthalpy runs. These data were taken to confirm that two transitions do, indeed, exist and that the two peaks observed are not an experimental aberration. After going through the two transitions during heating, the cooling was stopped after the liquid- β transition and before the β - α transition could take place. At this point heating was resumed, and only the β -liquid transition was observed before reaching the peak temperature. The sample was then cooled, and during this cooling both transitions were present. This type of run was used several times at high pressures, and in all cases similar results were obtained.

Figure 10. Results from a special DTA run. Top traces show sample-reference temperature differences. Bottom trace shows, from left to right: increasing temperature through the α - β and β -liquid transitions, decreasing temperature through the liquid- β transition, increasing temperature through the β -liquid transition, and decreasing temperature through both transitions.

The amplitude of the peaks that were recorded during the enthalpy runs were determined by the cut-and-weigh method. There is, of course, potential error in this method of determining peak amplitudes, but the results of cutting and weighing the same peaks twice indicated that errors were $<5\%$. Figure 11 shows the mass of the α - β and β -liquid transition peaks in arbitrary units for the Be transitions as a function of passes through the transition region.

Using a value of $2.855 \text{ Kcal/g}\cdot\text{m}$ for the transition enthalpy of Ag, values of 1.9 and $2.2 \pm 0.2 \text{ Kcal/g}\cdot\text{m}$ were obtained for the α - β and β -liquid transition enthalpies, respectively, for Be. The Loasby and Dearden values of 0.5 and $0.6 \text{ Kcal/g}\cdot\text{m}$ for these transitions differ by a factor of about 4; however, the ratios for the values are fairly close. From our work, the enthalpy ratio of α - β / β -liquid is 0.86 , whereas, the ratio from Loasby and Dearden is about 0.8 . The total enthalpy for both transitions from our work is $4.1 \text{ Kcal/g}\cdot\text{m}$ compared to $1.1 \text{ Kcal/g}\cdot\text{m}$ from

Loasby and Dearden. In their drop-calorimeter experiment, Kantor et al.³ obtained a value of $3.5 \text{ Kcal/g}\cdot\text{m}$ for the total enthalpy for the two transitions. This value is within 20% of the value determined in our study.

The large difference in the transition temperatures for Ag and Be are certainly a cause for concern. However, this difference alone does not explain the discrepancy between our results and those of Loasby and Dearden since an Ag standard was used in both studies. In an attempt to use a standard in which the transition temperature is closer to the Be transitions, Mn was run in our apparatus. The results were of no use in the determinations of Be enthalpies since the Mn reacted with the BeO of the sample holder. The outcomes from the Mn test do, however, point out some of the problems that can occur when even a small reaction takes place between the sample and a foreign substance. The DTA traces are shown in Fig. 12 for six heating cycles for Mn. These traces show a dramatic change in character

Figure 11. (a) The DTA peak for Ag used in the enthalpy determinations. (b) An example of the DTA peaks used for Be in the enthalpy determinations.

Figure 12. A series of six temperature cycles for the Mn sample shows the changing character of the DTA curves.

as the interaction between BeO and Mn took place. An x-ray analysis for the Mn sample, performed after the high-temperature runs, is shown in Table 3. After the six heating cycles, the only

notable change in the Mn was an increase from 2 to 500 ppm by weight Be. These Be values held for both the surface and bulk of the Mn.

Table 3. X-ray analysis for the Mn sample used in the enthalpy attempt.

Element ^a	Experimental Mn sample		Unused Mn sample (ppmw)
	Outside surface (ppmw)	Inside surface ^b (ppmw)	
B	500	500	500
Cu	300	300	300
Cr	150	150	100
Fe	150	150	150
Si	300	300	150
Ag	50	2	90
Ca	100	50	50
Al	5	5	5
Be	500	500	2
Mg	50	50	5
Pt	<30	<30	<30

^a The following elements were not detected: Sb < 500 ppmw; Ba, Bi, Cd, Nb, Pb, V, Zn, and Zr < 150 ppmw; Mo < 80 ppmw; Co < 50 ppmw; Ni < 20 ppmw; Ti < 5 ppmw; Sr < 2 ppmw.

^b Results for a surface exposed when the sample was cut in two.

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