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# *Influence of Magnesium on Some of the Physical Properties of Cerium*[**]

(1)

## ABSTRACT

The lattice parameters and low-temperature thermal expansion behaviors of a number of cerium-rich alloys containing up to 13 a/o magnesium have been examined. The lattice parameter data indicate a maximum solid solubility of 5.9 a/o magnesium in $\gamma$-Ce, and a positive deviation from Vegard's law. The dilation data show that magnesium decreases the temperature of the $\gamma$–$\alpha$ electronic transformation in cerium at the rate of about 11°K per 1 a/o magnesium. The composition dependence of the transformation temperature, the length change at the transformation and the coefficient of expansion indicate that a critical point would exist at about 10 a/o magnesium, 1 atm and 0°K, if this amount of magnesium were soluble in cerium. Magnesium is found to stabilize $\gamma$-Ce with respect to $\beta$-Ce; i.e., it tends to prevent the formation of the hexagonal phase. A comparison of these results with those reported earlier indicate that the $\alpha$–$\gamma$ transformation temperature depends on the average concentration of electrons in the valence band.

Some of the properties of the cerium stock used in this investigation are compared with those of cerium metal previously used in earlier studies.

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## INTRODUCTION

A recent study of the influence of alloying elements on the $\gamma$–$\alpha$ electronic transformation of cerium revealed that the valence of the solute is quite important in determining the relative stabilities of the $\gamma$-Ce and $\alpha$-Ce phases,[1] and it was shown that the higher the valence of the solute the greater is the tendency for the higher valent form ($\alpha$-Ce) to be stabilized. That study, however, was limited to investigating the effects of solutes having valences equal to or greater than three, which essentially is the valence of the lower valent form ($\gamma$-Ce). The main reason for making the present investigation was to see if the ideas and conclusions established for the higher valent solutes would also apply to those having valences less than three.

Only a few elements having a valence of one or two are expected to be soluble in cerium.[2,3] These are magnesium, calcium, ytterbium, europium, sodium and lithium. Experimental data indicate that magnesium is soluble in cerium,[2,4] calcium is insoluble,[2,5] and ytterbium and europium are soluble but appear to behave as if they were trivalent at concentrations below 1 or 2 a/o.[6] There are no reliable experimental data concerning the solubilities of sodium and lithium in cerium.

Vogel and Heumann[4] have given the most accurate description of the phase relationships in the cerium-rich portion of the cerium-magnesium phase diagram (figure 1). Their results indicate that about 24 a/o magnesium is soluble in the high-temperature body-centered cubic ($\delta$) form of cerium, but they presented no accurate data for the solubility of magnesium in the phase of interest in the present investigation; i.e., face-centered cubic $\gamma$-Ce.*

Mahn's low-temperature magnetic susceptibility data[7] indicate that at least 4 a/o magnesium is soluble in $\gamma$-Ce. Furthermore, her results also indicate that the $\gamma \rightarrow \alpha$ and $\alpha \rightarrow \gamma$ transformation temperatures decrease with increasing magnesium content and that the $\gamma$ phase does not transform to $\alpha$ in alloys containing more than about 5 a/o magnesium.

* The solvus line shown in figure 1 between $\gamma$-Ce and the two phases $\gamma$-Ce + CeMg at about 450° C is based on results obtained in this study.

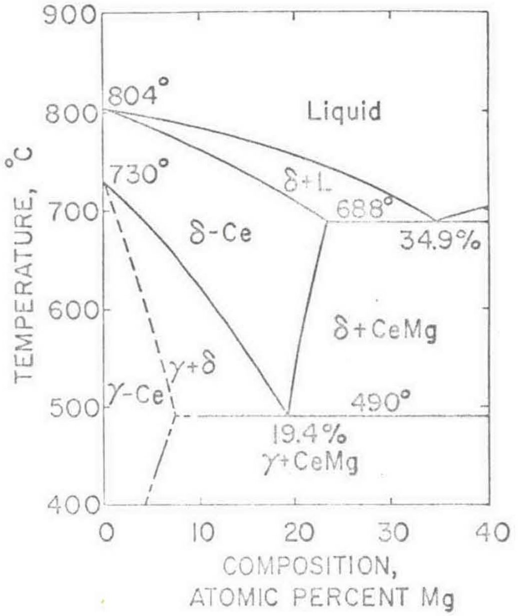

Fig. 1. Cerium-rich end of the cerium-magnesium phase diagram.

On the basis of the above information it was decided that the first step in this investigation would be to study the effect of magnesium additions on some of the physical properties of cerium. Since the paucity of reliable solid solubility data made it obviously necessary to obtain such data before attempting to interpret the low-temperature dilatometric data, the lattice constants of all alloys used in this investigation were measured to provide the needed solubility information.

## EXPERIMENTAL

The alloy specimens were prepared by melting together weighed amounts of cerium and magnesium in an induction furnace. The chemical analysis of the cerium stock used in this study is shown in Table I. The molten alloys were cast to form right cylinders 0.6 cm diameter by 2.5 cm long, and the castings were heat treated for a minimum of 200 hours at 450°C (723°K) and then air-cooled.

Since appreciable amounts of magnesium were lost during sample preparation it was necessary to analyze some of the specimens chemically to determine their compositions. The seven compositions given in figure 2 are based on chemical analysis.

Table I: Chemical analysis of cerium

| Impurity | (w/o) | (a/o) |
|---|---|---|
| hydrogen | 0.016 | 2.18 |
| carbon | 0.007 | 0.08 |
| nitrogen | <0.001 | <0.01 |
| oxygen | 0.008 ± 0.002 | 0.07 ± 0.02 |
| magnesium | 0.05 | 0.29 |
| manganese | 0.05 | 0.13 |
| copper | 0.001 | 0.02 |
| lanthanum | 0.05 | 0.05 |
| cerium[a] | 99.82 | 97.17 |

[a] Cerium content by difference.

The compositions of the other six specimens, which contained less than 6 a/o magnesium, were estimated from the lattice parameter-versus-composition curve (figure 2) in the single-phase $\gamma$-Ce region (this point is discussed later).

Filings taken from the sides of the heat-treated cylindrical specimens were sealed within evacuated capillary tubes and were further heat treated to remove any cold work. This second heat treatment consisted of holding the samples at 450° ± 10°C (723° ± 10°K) for 10 or 15 minutes and then quenching them in water. A 114.6 mm diameter Debye-Scherrer camera and Cu $K_\alpha$ radiation were [illegible] in obtaining the X-ray powder patterns. The lattice constants were determined from the back reflection region doublets by using a $\Phi \tan \Phi$ extrapolation method.[8]

The low-temperature dilatometric techniques[9] and the interpretation of the dilatometric-versus-temperature curves[10] are described elsewhere.

## RESULTS AND DISCUSSION

### X-ray data

*Pure Cerium.* The lattice parameter of the cerium stock used in this investigation is shown in Table II. Also shown in Table II are the data for two different cerium stocks used in previous studies.[1,6,10,11] It is interesting to note that there is essentially no difference in the lattice constants for these three materials, even though the total impurity contents vary quite widely. This is in contrast with the $\gamma \rightarrow \alpha$ transformation temperature and length change which are quite different for the three specimens.

Table II: A Comparison of Some of the Properties of the Various Cerium Stocks Used in This and Previous Studies

| Property | USBM–Ce[10] | This Study | CMC-Ce[10,11] |
|---|---|---|---|
| Purity (a/o) | 99.53 | 97.17 | 96.41 |
| Purity (w/o) | 99.94 | 99.82 | 99.54 |
| $\gamma$-Ce lattice constant (Å) | 5.1601 ± 2[a] | 5.1602 ± 2[a] | 5.1599 ± 3[a] |
| $\alpha$–$\gamma$ intermediate phase lattice constant (Å) | 5.1233 ± 5[a] | N.F.[b] | N.F.[b] |
| $\gamma \rightarrow \alpha$ trans. temp. (°K) | 116.3 ± 4.3 | 111 ± 2 | 106.4 ± 4.6 |
| $\alpha \rightarrow \gamma$ trans. temp. (°K) | 178.7 ± 4.2 | 179 ± 1 | 174.2 ± 3.8 |
| $\gamma \rightarrow \alpha$ trans. length change (per cent) | 3.40 ± 0.04 | 3.50 | 3.67 ± 0.20 |

[a] The shorthand notation is used here: 5.1601 ± 2 means 5.1601 ± 0.0002.

[b] N.F. means this phase was not found.

*$\alpha$–$\gamma$ Intermediate Phase.* Gschneidner and co-workers[10] found a new face-centered cubic phase, $a_0 = 5.1233$Å, for cerium in a very high purity cerium specimen (see Table II, USBM-Ce), but were unable to observe this phase in a much less pure cerium sample (see Table II, CMC-Ce). Since the lattice parameter of this new phase lies between that of $\gamma$-Ce, $a_0 = 5.1601$, and $\alpha$-Ce, $a = 4.85$, they called it the "$\alpha$–$\gamma$ intermediate phase." The cerium stock used in this study, which has a purity intermediate between those used earlier, was examined to see if the $\alpha$–$\gamma$ intermediate

phase could be obtained by proper cooling techniques. All attempts to produce this phase, however, were unsuccessful.

Although these results were negative, they may be helpful in determining the impurity or impurities which may be responsible for the different behaviors.

*Lattice Parameters of Cerium-Magnesium Alloys.* The lattice parameter-versus-composition data are shown in figure 2, where it is seen that magnesium decreases the lattice parameter of cerium until the solubility limit (corresponding to 450°C ± 10°C, 723° ± 10°K) of 5.9 a/o magnesium is reached. A complete listing of the lattice parameters of these alloys is given in Appendix I. At magnesium concentrations greater than 5.9 a/o, two phases co-exist, $\gamma$-Ce solid solution and the intermetallic compound CeMg (CsCl, *B2* type). In the two-phase region the weighted, mean lattice parameter of $\gamma$-Ce saturated with magnesium was found to be 5.1321 ± 0.0002 Å.

The solubility limit at 450° ± 10°C as determined from figure 2 was used to draw the solid solvus line between $\gamma$-Ce and the two phases $\gamma$-Ce + CeMg in figure 1. It should be noted that the slope of this line is not known. It was drawn with the positive slope shown in figure 1, since this slope agrees quite well with the construction proposed by Vogel and Heumann[4] for the cerium-magnesium phase diagram.

The lattice parameter-composition curve appears to be linear between 0.5 and 4.2 a/o and is reasonably assumed to remain linear between 4.2 and 5.9 a/o magnesium, the solid solubility limit. Because of its linearity and the large change in lattice parameter with composition (0.0049 Å per 1 a/o), this curve was used in determining the compositions of specimens that were not chemically analyzed. The uncertainty in the compositions determined by this technique is generally less than ±0.10 a/o (see Appendix I). The uncertainty in the composition was estimated in the following manner: (1) a change of 0.0001 Å in the lattice parameter is equivalent to a change of 0.02 a/o; (2) the uncertainty of the true location of the $a_0$-versus-composition line in the single phase region (figure 2) was estimated to be ±0.0002 Å or ±0.04 a/o; and (3) the uncertainty of the lattice parameter of the alloy, which is of the order of ±0.0003 Å or ±0.06 a/o, was added to that obtained in (2) to give the estimated error for the composition. Thus, it is seen that this technique afforded a rapid and reasonably accurate method of determining the compositions of the single-phase alloys.

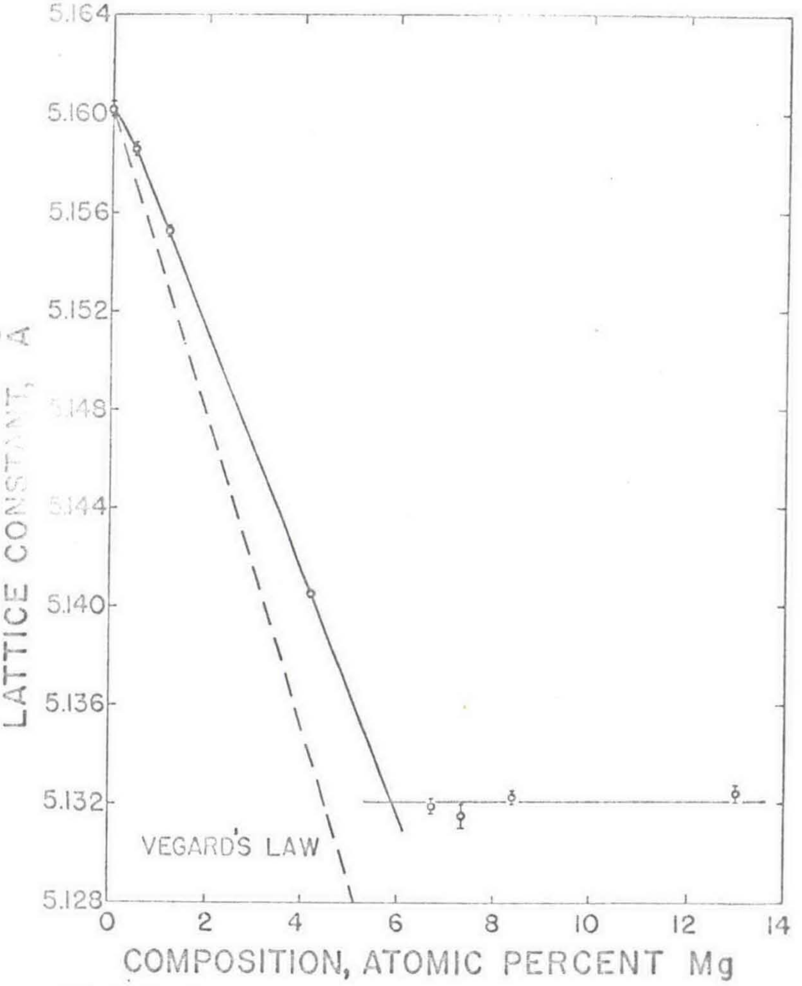

Fig. 2. Lattice parameters of some cerium-magnesium alloys as a function of composition.

*Deviation from Vegard's Law*. The single-phase cerium-magnesium alloys deviate positively from Vegard's law (see figure 2), and this departure was analyzed in terms of several different approaches that have been summarized by Gschneidner and Vineyard.[12] The approaches of Pines,[13] Fournet,[14] and Friedel[15] predict that these alloys will show negative deviations, and the approaches of Grum-Grzhimaylo,[16] Jaswon, Henry, and Raynor,[17] Zen,[18] Sarkisov,[19] and Gschneidner and Vineyard[12] predict a positive departure (figure 3). The approaches of Pines, Fournet, Friedel, and Gschneidner and Vineyard are generally restricted to alloys in which the two components have similar outer electronic structures. It would appear that these techniques should not be applied to cerium-magnesium alloys; however, the semi-empirical approach of Sarkisov indicates that the differences in the electronic configurations of cerium and magnesium accounts for less than one-tenth of the observed deviation. Because of this, we have also applied the four above mentioned techniques to these alloys.

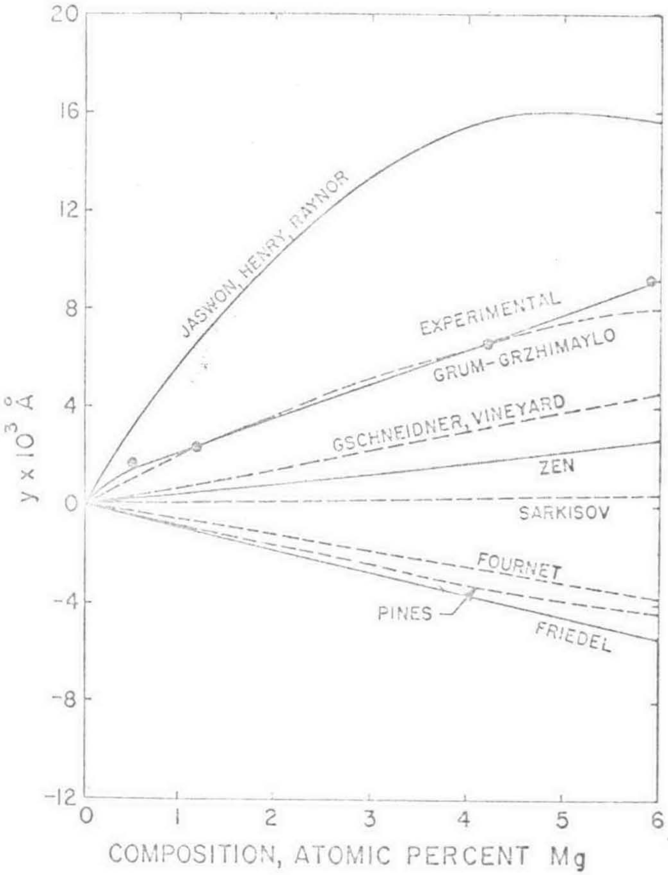

Fig. 3. Comparison of the observed departure from Vegard's law with several different theoretical and empirical approaches.

Of the five methods which indicate positive deviations, that of Grum-Grzhimaylo best describes the observed deviation. However, this is to be expected because use of this approach requires that two arbitrary constants be fitted to the experimental data. The technique of Jaswon, Henry and Raynor, which also requires knowledge of the lattice constants of the alloys, predicts a departure much larger than is actually observed. Of the remaining three approaches that predict a positive deviation, the method of Gschneidner and Vineyard agrees best with the experimental data.

## DILATOMETRIC DATA

*Pure Cerium*. The results listed in Table II show that the $\gamma \rightarrow \alpha$ transformation temperature and length change are quite sensitive to impurities, but that the $\alpha \rightarrow \gamma$ transformation temperature is not. This behavior is in agreement with the results obtained in our alloying studies previously reported.[1,6] The higher the total impurities the lower the transformation temperature, but the larger the length change. The latter is almost certainly due to the tendency of the impurities to prevent the formation of $\beta$-Ce. Further discussion at this time is not warranted because the kind and amounts of impurities vary from sample to sample. These data do, however, point out the need for a sufficiently large quantity of high purity cerium to determine the effects of small amounts of impurities on the properties of cerium, especially on the electronic transformation.

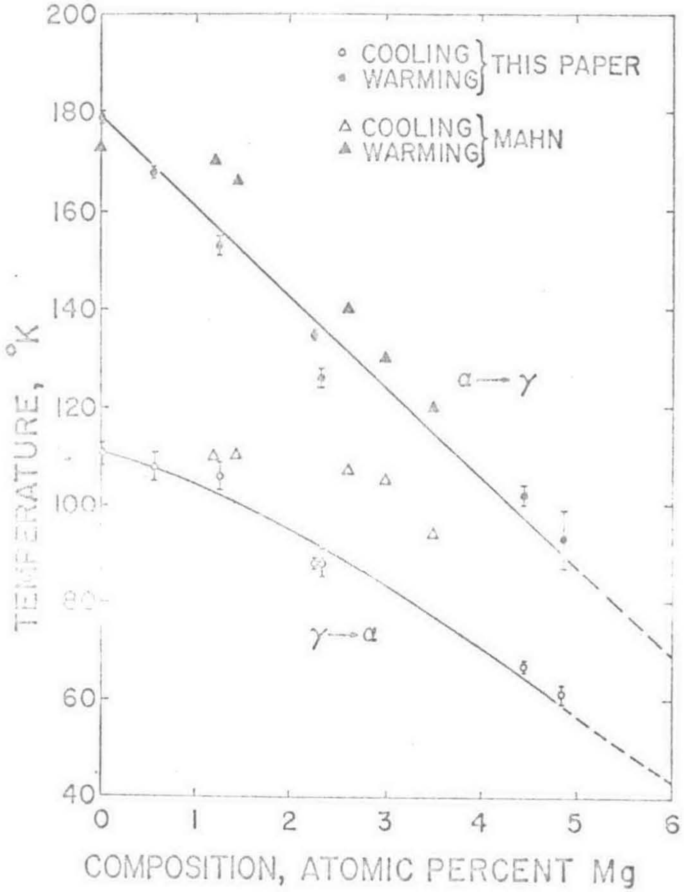

Fig. 4. The $\gamma \rightarrow \alpha$ and $\alpha \rightarrow \gamma$ transformation temperatures of some cerium-magnesium alloys as a function of composition.

*$\gamma$-$\alpha$ Transformation Temperatures.** The $\gamma \rightarrow \alpha$ and $\alpha \rightarrow \gamma$ transformation temperatures that were determined in the present investigation are shown in figure 4 along with Mahn's[7] transformation temperatures, which were determined from the reciprocal of the magnetic susceptibility-versus-temperature curves. The agreement between the two sets of data seems to be reasonable except

for the $\gamma \rightarrow \alpha$ transformation temperatures of the alloys containing more than 2 a/o magnesium. This discrepancy may be due in part to different kinds or amounts of impurities in the starting materials, and possibly to the difference between the experimental techniques used to measure the transition temperatures. The solid lines drawn in figure 4 are based on the data obtained in this investigation.

*Existence of a Critical Point.* Some evidence that suggests the existence of a critical point between 6 and 12 a/o magnesium was obtained. The average slopes of the transformation temperature-versus-composition curves, figure 4, from 0 to 5 a/o magnesium are −10.9° and −18.4°K per 1 a/o for the $\gamma \rightarrow \alpha$ and $\alpha \rightarrow \gamma$ transformations, respectively. It should be noted that the $\gamma \rightarrow \alpha$ transformation temperature-versus-composition curve is not linear between 0 and 3 a/o magnesium, but that it appears to be linear at compositions greater than 3 a/o. The slope of the linear portion of this curve is −13.3°K per 1 a/o. The intersection of these two transformation temperature curves is calculated to be 11.0 a/o magnesium and 23°K(−296°C) if the slope of the linear portion of the $\gamma \rightarrow \alpha$ curve is used, or 9.1 a/o magnesium and 12°K (−261°C) if the average value of this slope is used.

The length change* for the $\gamma \rightarrow \alpha$ transformation (figure 5), as taken from the dilatometric curves, was found to increase with increasing magnesium content up to about 1.5 a/o. At higher magnesium contents this effect was reversed and the volume change was found to decrease with increasing magnesium content. Although these data are suggestive of a critical point between 6.1 (solid line) and 11.1 (dashed line) a/o magnesium, the solubility limit at 5.9 a/o prevents obtaining experimental confirmation of this interpretation of the data. The value of the length change of the 4.83 a/o magnesium alloy may be too low because the transformation may not have gone to completion at the lowest temperature attainable (48°–50°K) in this study. Thus, the dash line is probably a more accurate representation of the true behavior of these alloys than the solid line.

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* The transformation temperatures are listed in Appendix II.

* These data are tabulated in Appendix II.

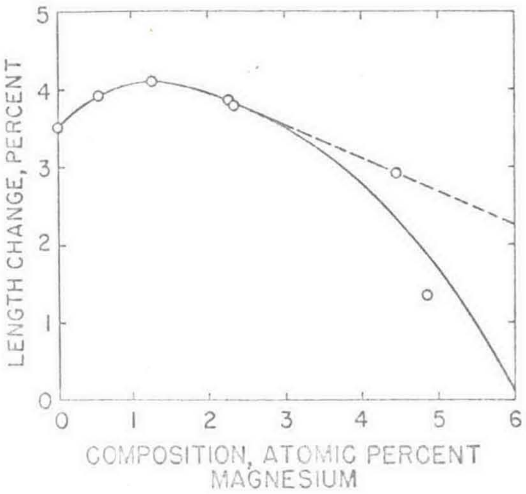

Fig. 5. The length change for the $\gamma \rightarrow \alpha$ transformation of some cerium-magnesium alloys as a function of composition.

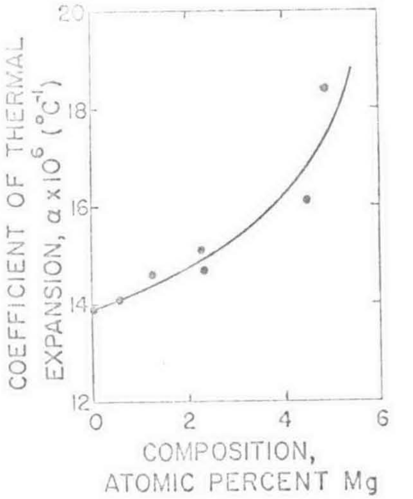

Fig. 6. Mean coefficient of linear thermal expansion from 200° to 295°K (−73° to +22°C) for some cerium-magnesium alloys as a function of composition.

These data, which are also substantiated by the thermal expansion results (see below), suggest that a critical point would exist at about 10 a/o magnesium, 0°K and 1 atm if this amount of magnesium were soluble in cerium.

*Expansion Coefficients.* The average coefficient of linear thermal expansion from 200° to 295°K (−73° to +22°C) is increased from $13.9 \times 10^{-6}$ (°K$^{-1}$) for pure cerium to $18.4 \times 10^{-6}$ (°K$^{-1}$) for the alloy containing 4.8 a/o magnesium (see figure 6 and Appendix III). The increasing value of the thermal expansion coefficient with increasing magnesium content is also indicative of an impending critical point at higher magnesium concentrations. Waber and co-workers[20] have found in cerium-thorium alloys that the coefficient of expansion is quite large, $\sim 60 \times 10^{-6}$, for the alloys in the vicinity of the critical composition. One might reasonably expect the coefficient of expansion to reach this value for the critical composition of these cerium-magnesium alloys. On the basis of this assumption and the data shown in figure 6 it is concluded that the critical composition is probably higher than 6 a/o magnesium. These data, therefore, also support the choice of 10 a/o magnesium for the critical composition rather than the lower value of about 6 a/o magnesium.

*Formation of β-Ce.* As is well known, β-Ce (hex. 2*c*-axis) forms from γ-Ce at approximately 250°K (−23°C) during cooling.[10] In previous studies it was observed that the solutes scandium, thorium and plutonium stabilize γ-Ce, and thus prevent β-Ce from forming, if more than 10 a/o of the solute is present,[1] and that the rare earths, in general tend to stabilize β-Ce.[6] It is possible to detect the formation of β-Ce from the appearance of the dilatometric curve, even though the volume difference between β and γ is only 0.4 percent and only small percentages (<10 percent) of β may be present.[1,6] Examination of the dilatometric curves for the cerium-magnesium alloys indicates that the amount of β-Ce formed decreases with increasing magnesium content. That is, 30 percent of β-Ce formed in pure cerium during the first cooling cycle was decreased to about 5 per cent in the 4.4 a/o magnesium alloy. Therefore, it is concluded that magnesium is a γ-Ce stabilizer.

*Influence of Valency.* It was mentioned in the Introduction that the primary reason for making this investigation of cerium-magnesium alloys was to determine if the valency effect[1] previously observed for solutes having valences greater than three, where the solvent is cerium, would also be found in solutes having valences less than three. It had been found[6] that, if a size correction were made for the rare earth solutes that have no unpaired 4*f* electrons, the $\gamma$–$\alpha$ transformation temperature for an alloy containing 2 a/o rare earth solute would be essentially the same as that of pure cerium. Furthermore, the data for cerium alloys containing 2 a/o thorium (valence = four) and plutonium (valence = 4.8) indicate that thorium raises the transformation temperature of pure cerium a few degrees and plutonium raises it by almost 15°K[1] (see figure 7). As noted earlier magnesium, which is divalent, lowers the transformation temperature. The data shown in figure 7 indicate that the $\gamma$–$\alpha$ electronic transformation in pure cerium depends upon the valence of the solute. That is, as the number of electrons in the valence band increases the $\alpha$-Ce phase becomes more stable with respect to the $\gamma$-Ce phase and, thus, the transformation temperature increases. Previous studies[1,6] had also shown that the solute atom's size and its number of unpaired 4*f* electrons play an important role in this respect.

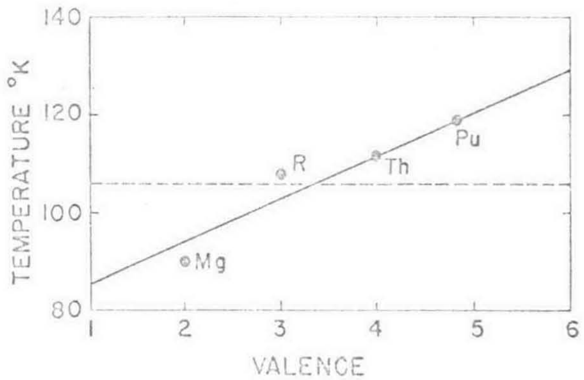

Fig. 7. Influence of the valence of the solute on the $\gamma \rightarrow \alpha$ transformation temperature of cerium for 2 a/o solute additions. The dashed line represents the transformation temperature of pure cerium. The data presented in this figure are normalized to the transformation temperature of the CMC–Ce stock (see Table II).

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## APPENDIX I

Lattice Parameter of Cerium-Rich Cerium-Magnesium Alloys

| Composition (a/o Mg) | | Lattice Constant |
|---|---|---|
| Nominal | Analyzed | (Å) |
| 0.0 | — | 5.1602 ± 2[a] |
| 1.0 | 0.52 | 5.1586 ± 2 |
| 1.0 | 0.55 ± 0.08[b] | 5.1584 ± 2 |
| 2.0 | 1.19 | 5.1553 ± 2 |
| 2.0 | 1.24 ± 0.08[b] | 5.1550 ± 2 |
| 3.5 | 2.26 ± 0.08[b] | 5.1500 ± 2 |
| 2.5 | 2.32 ± 0.08[b] | 5.1497 ± 2 |
| 4.0 | 4.20 | 5.1405 ± 1 |
| 4.0 | 4.44 ± 0.10[b] | 5.1393 ± 3 |
| 6.0 | 4.83 ± 0.12[b] | 5.1374 ± 4 |
| 7.0 | 6.75 | 5.1319 ± 3 |
| 8.0 | 7.36 | 5.1315 ± 5 |
| 10.0 | 8.42 | 5.1323 ± 2 |
| 14.0 | [illegible] | 5.1324 ± 3 |

[a] The shorthand [illegible] is used here to report the errors: i.e., 5.1602 ± 2 means 5.1602 ± 0.0[illegible]

[b] Composition determined from the lattice spacings versus composition curve (figure 2). The error listed for these compositions is that due to the error in the measured lattice constants (see text).

## APPENDIX II

Transformation Temperatures and the $\gamma \rightarrow \alpha$ Transformation Length Changes

| Analyzed Composition (a/o Mg) | Transformation Temperature | | Length Change (o/o) |
|---|---|---|---|
| | $\gamma \rightarrow \alpha$ (°K) | $\alpha \rightarrow \gamma$ (°K) | |
| pure Ce | 111 ± 2 | 179 ± 1 | 3.50 |
| 0.55 | 108 ± 3 | 168 ± 1 | 3.92 ± 0.02 |
| 1.24 | 106 ± 3 | 153 ± 2 | 4.10 |
| 2.26 | 88 ± 1 | 135 ± 2 | 3.88 |
| 2.32 | 88 ± 1 | 126 ± 2 | 3.80 |
| 4.44 | 67 ± 1 | 102 ± 2 | 2.93 ± 0.02 |
| 4.83 | 61 ± 2 | 93 ± 6 | 1.35 |

## APPENDIX III

Mean Coefficient of Linear Thermal Expansion from 200° to 295°K (−73° to 22°C)

| Analyzed Composition (a/o Mg) | Coefficient of Expansion $\bar{\alpha} \times 10^6$ (°C$^{-1}$) |
|---|---|
| pure Ce | 13.9 |
| 0.55 | 14.1 ± 0.8 |
| 1.24 | 14.6 ± 3.0 |
| 2.26 | 15.1 ± 4.6 |
| 2.32 | 14.7 |
| 4.44 | 16.1 ± 0.2 |
| 4.83 | 18.4 ± 1.0 |