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THERMAL DIFFUSIVITIES OF LANTHANUM, CERIUM AND PLUTONIUM AT HIGH TEMPERATURES

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ABSTRACT

The thermal diffusivities of La, Ce and Pu were measured in the high temperature regions which spanned their respective fcc, bcc and liquid phases. The transient heating technique was evaluated by successful measurements of the thermal diffusivity of liquid lead. The calculated thermal conductivity values for liquid La and Ce were the same as or less than their respective high temperature solid phases, while liquid Pu was a better conductor than any of its solid phases. The Lorenz numbers for liquid La and Ce were 10 and 30%, respectively, less than the free-electron value.

INTRODUCTION

The rare earth and actinide series of metals represent unique elements in the periodic table because of the appearance of *f*-orbital electrons. Recently, Gschneidner[1] has proposed the existence of two types of 4*f*-electrons in the rare earth metals, those in the atomic or "core" orbitals and those in the conduction or valence bands together with the 6*s*, 5*p* and 5*d* electrons. The physical properties, such as crystal structure, of the rare earth metals are affected by the 4*f*-electronic contributions to the valence band, which reach a maximum for Ce but are significant for La, Pr, Nd and Pm. Similarly, Hill[2] has shown by use of band structure calculations that the 5*f*-like fraction of the electronic charge reaches a maximum for U, Np and Pu in the actinide series.

For an evaluation of the transport behavior of the conduction electrons in these metals, this study was initiated to measure the thermal conductivities of La, Ce and Pu at sufficiently high temperatures to span their respective solid and liquid phases. Previous thermal conductivity values have been reported to 18 °C for La and Ce[3] and at 25 °C for Pu[4]. Because thermal conductivity of a metal is predominantly regulated by the flow of electrons, the thermal conductivity values are compared with reported electrical conductivity values by means of the Lorenz number.

A thermal diffusivity technique was utilized for these measurements. Thermal conductivity values were calculated from the thermal diffusivity measurements and

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accepted values for the density and heat capacity. The thermal diffusivity of liquid lead was determined in the apparatus in order to assess the accuracy of the technique.

EXPERIMENTAL

A. The method

When a material initially at thermal equilibrium with its surroundings is disturbed by a temperature change, a temperature wave transits the material. During this transient state, the rate at which the temperature wave travels is dependent upon the thermal diffusivity of the material. For a homogeneous isotropic material the thermal diffusivity is defined by the relationship:

$$D = \frac{\lambda}{dC} \text{ cm}^2 \text{ sec}^{-1} \tag{1}$$

where D = thermal diffusivity, C = heat capacity per gram, λ = thermal conductivity and d = density. If the thermal conductivity in the material is constant over the temperature range being considered, the conduction of heat is expressed in cylindrical coordinates by the relationship[5]:

$$\frac{\delta^2 T}{\delta r^2} + \frac{1}{r}\frac{\delta T}{\delta r} + \frac{1}{r^2}\frac{\delta^2 T}{\delta \phi^2} + \frac{\delta^2 T}{\delta z^2} = \frac{1}{D}\frac{\delta T}{\delta t} \tag{2}$$

where T = temperature and t = time.

In this experiment the apparatus was designed so that angular symmetry in temperature distribution was assumed and axial temperature gradients were eliminated. As a result, eqn (2) becomes:

$$D\left[\frac{\delta^2 T}{\delta r^2} + \frac{1}{r}\frac{\delta T}{\delta r}\right] = \frac{\delta T}{\delta t} \tag{3}$$

which is the desired heat flow equation for a circular cylinder with only a radial temperature gradient.

A solution of eqn (3) for the transient case is possible when the experimental condition is imposed that the temperatures at all points in the cylinder change at the same constant rate. This condition is achieved experimentally when the surface temperature of the cylinder is increased at a uniform rate so that the term $\delta T/\delta t = q$, a constant. An initial temperature disturbance occurs within the cylinder as soon as external heat is applied to the surface. This disturbance must disappear before meaningful data are accumulated. When the specified experimental conditions are satisfied, solutions of eqn (3) are possible and depend upon the boundary conditions.

Before the boundary conditions can be stated, the physical arrangement of the diffusivity container needs to be specified. Because the operating temperatures were above the melting points of these metals, the samples were encapsulated in cylindrical tantalum containers, 7.6 cm long. One thermocouple was inserted in a thermowell located in the outer wall of the container. A second thermocouple was located in the center of a tantalum bob which was located along the axis of the container. The temperature difference ΔT between the two thermocouples was determined as the sample was heated externally at a constant rate. The dimensions of importance are: r_1 = the o.d. radius of the inner bob; r_2 = the i.d. radius of the outer wall; and r_3 = the distance from the center of the cylinder to the center of the outer thermocouple.

When long, solid cylinders are used, Ginnings[6] showed that for the ideal case where there is no material inside r_1, and when no heat loss occurs across the r_1 surface, a solution for eqn (3) is

$$\Delta T = \frac{1}{4D} q[r_2^2 - r_1^2(1+2\ln r_2/r_1)]. \tag{4}$$

The present diffusivity container does not fit this case because heat loss does occur both at the r_1 and r_2 surfaces, and the total ΔT is composed of temperature differences across the center bob, the sample layer and the wall. The solution of eqn (3), for this case is,

$$\Delta T = q\left[\frac{C_b r_1^2}{2}\left(\frac{\ln\frac{r_2}{r_1}}{\lambda_s} + \frac{\ln\frac{r_3}{r_2}}{\lambda_w}\right) + \frac{r_1^2}{4D_b} + \frac{C_s}{2}(r_2^2 - r_1^2)\left(\frac{\ln\frac{r_3}{r_2}}{\lambda_w}\right) + \right.$$
$$\left. + \frac{1}{4D}\left[r_2^2 - r_1^2\left(1+2\ln\frac{r_2}{r_1}\right)\right] + \frac{1}{4D_w}\left[r_3^2 - r_2^2\left(1+2\ln\frac{r_3}{r_2}\right)\right]\right] \tag{5}$$

where C_s = heat capacity of sample per unit volume, C_b = heat capacity of bob per unit volume, λ_s = thermal conductivity of sample (cal sec^{-1} cm^{-1} deg^{-1}), λ_w = thermal conductivity of wall (cal sec^{-1} cm^{-1} deg^{-1}), D_b = thermal diffusivity of the bob, D_w = thermal diffusivity of the wall. No consideration has been allowed in eqn (5) for possible thermal resistances at the two sample to container interfaces. When the sample is liquid, good thermal contact should be achieved between the liquid and the tantalum container. When the sample is resolidified, good thermal contact is dependent upon the absence of any gaps forming between the sample and the container.

Chromel–alumel thermocouples, sheathed in stainless steel, were inserted into the thermowells of the diffusivity container. The temperature difference was determined from the potential difference between these two thermocouples. This potential difference was amplified 50 times and plotted on the X-axis of an electronic X–Y recorder. The thermocouple in the center of the diffusivity container controlled the Y-axis of the recorder which plotted the sample temperature.

An absolute determination of ΔT would require an accurate evaluation of the e.m.f. readings from each thermocouple over a wide temperature range. In order to avoid this lengthy determination, an initial technique attempted to calibrate the thermocouples when they were inserted in a silver cylinder for which ΔT was nearly zero. Subsequent experimentation showed, however, that the replacement of the thermocouples into the diffusivity container often affected the potential difference. For this reason during subsequent determination, the thermocouples remained in the diffusivity container and the measurements were made in a differential manner. In the differential technique, the temperature difference ΔT was determined between two successive heating excursions made at two different heating rates and q in eqn (5) became Δq. The values of q utilized were: 0.256, 0.206, 0.143, and 0.0838 °C sec^{-1}, although not all heating rates were used for every determination.

This latter technique was used to check the previously reported results[7] obtained with Pu measured by the former technique. A new sample was used for these latter measurements reported here.

The diffusivity container was placed vertically in a tube furnace. The primary heating element of the furnace was connected to a program-controlled power supply. Guard heaters above and below the primary heating element were controlled by a differential temperature system so that they remained within $\pm 2°$ of the primary heater. The entire furnace operated within a hermetically sealed enclosure which was evacuated and filled with helium in order to prevent oxidation of the tantalum diffusivity container.

B. Materials

All of the specimens were prepared from high-purity materials. The La and Ce were obtained from Research Chemicals Division of Nuclear Corporation of America. The spectrographically detectable impurities in these samples (in wt %) were: 0.01 Y, 0.01–0.05 Si, 0.01–0.02 Fe, 0.01 Mg, 0.01 Al, and 0.03 Ca. The lead obtained from American Smelting and Refining Company had a quoted purity of 99.999 wt %. The Pu, prepared by electrorefining techniques at the Rocky Flats Division of the Dow Chemical Company, had as major impurities (in ppm by weight): Am-46, U-9, Fe-11, Ta-19, W-67, C-32 and O_2-69.

Each specimen was prepared in the shape of a right-circular cylinder containing an axial hole to accommodate the central thermocouple bob. They were loaded into the bottom of the diffusivity containers in an inert atmosphere. The end caps were placed on the containers and sealed by either electron-beam or tungsten–inert gas

TABLE 1

DIMENSIONS OF THERMAL DIFFUSIVITY SPECIMENS

Metal	*Capsule radii (cm)*			*Sample weight (g)*
	r_1	r_2	r_3	
La	0.159	0.757	0.916	65.2
Ce	0.159	0.757	0.916	75.5
Pu	0.209	0.826	0.976	227.0
Pb	0.159	0.757	0.916	110.3

welding techniques. Each filled container was heated above the melting point of the specimen and cooled to ambient temperature in order to obtain contact between the container and the specimen. The dimensions of the specimens are given in Table 1.

RESULTS

Each specimen was heated successively at several heating rates. The value of ΔT changed only slightly as each heating excursion transited the stable temperature range of each metallic allotrope. At the phase transformation temperature, however, ΔT initially decreased and then increased to form a large peak on the recorder trace, as described by Sze and Meaden[8] for all first-order phase transformations. The observation was made that the presence of these two extrema indicated that good thermal contact had been achieved between the sample and the container so that reliable thermal diffusivity values were obtained.

The reproducibility of the chromel–alumel thermocouples was $\pm 0.5\,\mu$V for two successive heating excursions at the same heating rate; consequently, for successive determinations made at two heating rates, the possible deviation would be $\pm 1.0\,\mu$V or $\pm 0.02\,^{\circ}$C. This deviation represented possible errors of ±10% for liquid Pb, ±18% for La and Ce and ±8% for the Pu determinations. Because this was the largest error in the measurements, it forms the basis for the deviations given in the data.

The average values for the thermal diffusivity, D, and the corresponding calculated values for the thermal conductivity, λ, are reported, Table 2. Based on the assumption that the lattice contribution to the thermal conductivity is insignificant for these metals in this temperature range, the thermal conductivity, λ, and the electrical resistivity, ρ, are compared by use of the Lorenz number, defined as,

$$L = \lambda\rho/T$$

where $T = {}^{\circ}\mathrm{K}$ and

$$L = 2.4 \times 10^{-8}\ \mathrm{W\ ohm\ deg^{-2}}\ \text{for a free-electron metal.}$$

The electrical resistivities have been reported for all of these metals in the temperature range of interest, except for liquid Pu.

Liquid lead

The thermal conductivity values calculated for liquid lead between 450 and 650 °C, Fig. 1, show an increase with temperature. The large increase in the thermal conductivity between 550 and 600 °C is attributed to the onset of convective flow which caused a noticeable decrease in the recorded differential temperatures and should, therefore, be discounted.

A number of values have been reported for the thermal conductivity of liquid lead. The data selected for Fig. 1 are typical examples. The values reported by Powell and Tye[9], curve 1, increase with temperature while the most recent data by Duggin[10], curve 3, decrease with temperature. A determination based upon thermal diffusivity

TABLE 2

THERMAL DIFFUSIVITY, CONDUCTIVITY AND LORENZ NUMBERS FOR LANTHANUM, CERIUM AND PLUTONIUM

Temperature (°C)	*Phase*[a]	*D* *cm*^{2} *sec*$^{-1}$	*d* *g cm*$^{-3}$	*C* *cal g-at*$^{-1}$ *deg*$^{-1}$	*λ* *W m*$^{-1}$*deg*$^{-1}$	*ρ* *10*^{6} *ohm cm*	*Lorenz no.* *10*^{8} *W ohm deg*$^{-2}$
Lanthanum			(Ref. a, b)	(Ref. c)		(Ref. d, e)	
700	fcc	0.12 ±0.02	6.12	7.90	17±3	105	1.8±0.3
800	fcc	0.10 ±0.02	6.10	8.25	14±3	110	1.4±0.3
875	bcc	0.10 ±0.02	5.97	9.45	18±3	128	2.0±0.4
1000	liq.	0.12 ±0.02	5.94	8.20	18±3	144	2.1±0.3
Cerium			(Ref. a, b)	(Ref. f)		(Ref. g)	
200	fcc	0.12 ±0.02	6.76	7.00	16±3	90	3.1±0.6
300	fcc	0.12 ±0.02	6.73	7.37	18±3	95	3.0±0.6
400	fcc	0.12 ±0.02	6.72	7.74	19±4	100	2.8±0.5
500	fcc	0.12 ±0.02	6.71	8.14	20±4	104	2.6±0.5
600	fcc	0.14 ±0.02	6.69	8.57	24±4	108	3.0±0.5
700	fcc	0.08 ±0.02	6.67	9.02	14±3	110	1.6±0.3
770	bcc	0.10 ±0.02	6.70	9.05	18±4	120	2.1±0.4
850	liq.	0.08 ±0.02	6.67	9.35	16±3	127	1.8±0.3
875	liq.	0.08 ±0.02	6.66	9.35	15±3	128	1.7±0.3
Plutonium			(Ref. h)	(Ref. h)		(Ref. h)	
250	fco	0.055±0.004	17.14	8.60	14±1	107.8	2.9±0.2
300	fco	0.058±0.005	17.14	8.60	15±1	107.8	2.8±0.2
350	fcc	0.050±0.004	15.92	9.00	13±1	100.4	2.1±0.2
400	fcc	0.054±0.004	15.92	9.00	14±1	100.4	2.1±0.2

TABLE 2 (*continued*)

Temperature (°C)	*Phase*[a]	*D* $cm^2\ sec^{-1}$	*d* $g\ cm^{-3}$	*C* $cal\ g\text{-}at^{-1}\ deg^{-1}$	λ $W\ m^{-1}\ deg^{-1}$	ρ $10^6\ ohm\ cm$	*Lorenz no.* $10^8\ W\ ohm\ deg^{-2}$
450	fcc	0.054±0.004	15.92	9.00	14±1	100.4	2.0±0.2
500	bcc	0.033±0.003	16.51	8.40	8±1	110.6	1.1±0.1
550	bcc	0.034±0.003	16.51	8.40	9±1	110.6	1.2±0.1
600	bcc	0.038±0.003	16.51	8.40	9±1	110.6	1.1±0.1
675	liq.	0.057±0.005	16.60	10.00	17±1		
700	liq.	0.057±0.005	16.57	10.00	17±1		
750	liq.	0.056±0.005	16.49	10.00	16±1		
800	liq.	0.056±0.005	16.41	10.00	16±1		

[a] fco = face-centered orthorhombic; fcc = face-centered cubic; bcc = body-centered cubic; liq = liquid.

References for Table 2

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by Yurchak and Filippov[11], curve 2, is closer to the values of curve 1 but decreases with temperature. The present values between 450 and 550 °C lie within the range of the previous experimental values and increase with temperature. All of the experimental values appear to be less than the values calculated by the Lorenz function (curve L) based upon the electrical conductivity.

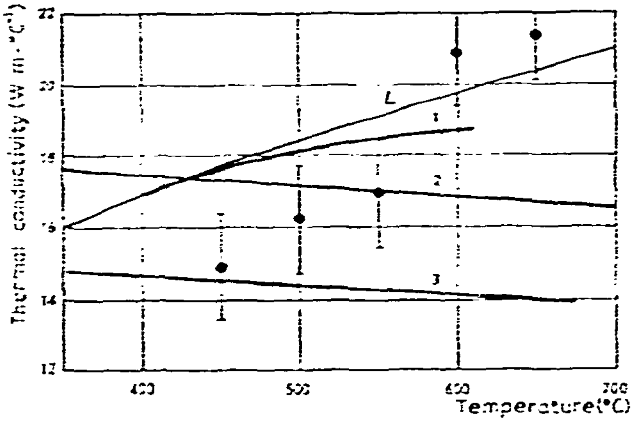

Fig. 1. Comparison of the thermal conductivity values of liquid lead. L = Lorenz function based upon electrical conductivity; (1) Powell[9]; (2) Yurchak[11]; (3) Duggin[10]; ●, present data.

Lanthanum

The heating traces for La were not reproducible until the temperature reached approximately 700 °C. Apparently, at this temperature the lanthanum had expanded sufficiently so that good thermal contact was achieved between the sample and the container. The thermal diffusivity values at temperatures between 700 and 1000 °C were reproducible except for the 800° value which was noticeably affected by the nearness of the fcc → bcc phase transformation. The reported thermal conductivity value of 17 ± 3 W m^{-1} deg^{-1} at 700 °C represents a gradual increase from the reported value[3] of 14 W m^{-1} deg^{-1} at 18 °C, although it is somewhat less than the value of 23 W m^{-1} deg^{-1} predicted by Ho, Powell, and Liley[12]. The value of D at 1000 °C, 0.12 ± 0.02 cm^2 sec^{-1}, compares very favorably with the exclusively liquid phase measurement of 0.11 ± 0.01 cm^2 sec^{-1} recently reported by Atalla[13]. Because of an unusually large value for heat capacity utilized by Atalla, a comparison of λ values cannot be made.

Cerium

The thermal diffusivity measurements appeared to be reliable from 200 to 875 °C, except for the value at 700 °C in which the recorded traces were affected by the nearness of the fcc → bcc phase transformation. A preliminary report[14] from this laboratory quoted thermal diffusivity measurements to nearly 1000 °C. Subsequently, it was determined that after each high temperature excursion the melting point increased until it had risen from 800 to nearly 830 °C. Because this observation might have indicated Ta contamination of the sample, the sample was discarded and a new sample obtained for this study. This latter specimen was heated to only 900 °C for a brief period of time and no effect upon the melting point was detected.

The thermal conductivity of the fcc phase apparently increases gradually from a reported value[3] of 11.2 W m^{-1} deg^{-1} at 18 °C up to 20±4 W m^{-1} deg^{-1} at 500 °C. The measured value at 500 °C is in good agreement with the value of 19 W m^{-1} deg^{-1} predicted by Ho, Powell and Liley[12]. The thermal conductivity decreases slightly in the bcc phase and then decreases further in the liquid phase. The reported thermal diffusivity of the liquid near the melting point, 0.08±0.02 cm^2 sec^{-1}, is somewhat lower but within the experimental error of the value, 0.10±0.01 cm^2 sec^{-1}, reported by Atalla[13] from measurements performed only in the liquid phase. Because of an abnormally large value used by Atalla for the density of the liquid, the thermal conductivity values cannot be compared. Also, the effect of contamination by tantalum upon the thermal conductivity of liquid Ce held at high temperatures for long periods of time in tantalum containers needs to be investigated in the method used by Atalla because of the effect upon the melting point of Ce noted previously.

Plutonium

The experimental procedure was altered slightly during the Pu measurements in order to avoid the large volume change associated with the $\alpha \rightarrow \beta$ phase transformation at 120 °C. After each heating excursion the sample was cooled to only 200 °C rather than to ambient temperature; consequently, The Pu transformed to the γ (face-centered orthorhombic) phase before a new heating excursion was initiated.

No data appear in the literature concerning the thermal conductivity of Pu in this temperature range. The present data indicate that the thermal conductivities of the γ and δ (fcc) phases are nearly the same at 13±1 W m^{-1} deg^{-1} and nearly twice the value[4] of 6.7 W m^{-1} deg^{-1} reported for the monoclinic α-phase at 25 °C. These trends are in qualitative agreement with the electrical resistivity measurements referenced in Table 2 for the α, γ and δ phases. The large decrease in the thermal conductivity of the ε (bcc) phase, Table 2, is not expected from comparison with the electrical resistivity measurements. It may be due, however, to an experimental difficulty caused by the volume contraction during the $\delta \rightarrow \varepsilon$ phase transformation so that good thermal contact was not maintained during the measurement of the ε phase. The large increase in the thermal conductivity at the melting point to 17 W m^{-1} deg^{-1} indicates that the liquid phase has a higher conductivity than any of the solid allotropes. Although this may be surprising, it is in good agreement with the value[15] of 17 to 19 W m^{-1} deg^{-1} measured between 420 and 520 °C for the highly concentrated Pu liquid alloy, 90.5 at % Pu–9.5 at % Fe.

DISCUSSION

Comparisons of the electrical resistivity and thermal conductivity changes at the melting points in Table 3 indicate that they are of the same order of magnitude and very small. In this regard they are similar to the transition metals, Fe, Co, Ni, Pt, W

TABLE 3

ELECTRICAL RESISTIVITY, THERMAL CONDUCTIVITY AND VOLUME[a] CHANGES AT THE MELTING TEMPERATURE

Metal	$\Delta\rho/\rho$	$\Delta\lambda/\lambda$	$\Delta V/V$
La	0.11	0	0.006
Ce	0.07	0.11	−0.010
Pu	n.a.[b]	−0.22[c]	−0.024

[a] L. J. Wittenberg and R. DeWitt, *J. Chem. Phys.*, 56 (1972) 4526. [b] n.a. = not available. [c] Based upon δ and liquid Pu.

and Mo which also have high electrical resistivities (~100 μohm cm) comparable to La, Ce and Pu. As discussed by Mott[16] the enhanced resistivity of crystalline Co, Ni and Pd must be related to the presence of "holes in the *d* bands" and that two separate mean free paths must be introduced for the liquid also. The fact that the valence bands of La and Ce contain 4*f* and 5*d*-like electrons, as calculated by Gschneidner[1], further supports the correlation that these two metals are transition-like metals in their behavior.

The slight volume decrease in Ce upon melting (Table 3) was explained by Jayaramann[17] as due to the transfer of an *f*-orbital "core" electron to the valence band. In this regard, the volume change at the melting point would be similar to the $\gamma \to \alpha$ Ce phase transformation which occurs at 7 kbar (25 °C) with a volume decrease of nearly 20%. Magnetic susceptibility measurements[18] indicate a decrease from 20×10^{-4} to 6×10^{-4} emu g-atom^{-1} during the $\gamma \to \alpha$ phase transformation at ambient temperature. On the other hand, magnetic susceptibility measurements at zero pressure decrease smoothly as a function of temperature with only a slight change of slope occurring at the melting point[19], and a value of the liquid phase nearly identical to the γ-phase under pressure. In regard to electrical conductivity, the electrical resistivity[20] decreases nearly 50% during the $\gamma \to \alpha$ phase transformation at 7 kbar. Conversely, electrical and thermal conductivity measurements indicate, Table 2, that the conductivity of the liquid phase is less than the solid phase. Electrical resistivity, thermal conductivity and magnetic susceptibility measurements, indicate, therefore, that the phase transformation at the melting point is not similar to the $\gamma \to \alpha$ phase transformation, and the *f*-electron character of the liquid is little changed from the solid phase. As a result, no simple mechanism exists to explain the volume contraction during melting.

The thermal conductivity of Pu increases nearly 22%, Table 3, during melting and has a much larger volume contraction than Ce. The volume contraction of Pu during melting is similar to that of Bi; however, the thermal conductivity[21] of Bi increases nearly 90% upon melting. Clearly, Pu at the melting point is not like a semi-metal. Apparently, 6*d* and possibly 5*f*-band scatter is important in both solid and liquid Pu, but a change in the electronic structure does occur at the melting point, similar to the $\gamma \to \alpha$ Ce phase transformation.

Several reports indicate that the Lorenz numbers of liquid Ga, Pb and In[10] decrease with increasing temperature until they are significantly below the free-electron value, although no such decrease was found for liquid Ga by Busch *et al.*[22]. The present data, Table 2, strongly indicate that the Lorenz numbers for liquid La and Ce are less than the free-electron value. Before this observation can be related to the behaviour of liquid Ga, Pb and In, it should be noted that Ce is one of the very few liquid metals with a positive Hall coefficient[19], while it is negative and near the free-electron value for liquid Ga, Pb and In[21]. A positive Hall coefficient for liquid Ce implies a non-spherical Fermi surface[16] and this effect upon the Lorenz number has not been evaluated.

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