

Electrical resistivity of La

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The electrical resistivity of high-purity double hexagonal-close-packed (dhcp) α -La from 5 to 300 K is reported. Measurements were made on small-grained samples prepared by heat treatment of cold-worked lanthanum. Measurements were also made on samples cut in different directions from an ingot slowly cooled from the molten state. The room-temperature results were all within 2% of the mean value. Chemically pure β -La (fcc) cannot be retained at room temperature, hence, measurements were made on an fcc sample of La containing 0.2-at. % Gd and ~ 0.8 -at. % total interstitial nonmetallic impurities. The cubic form has almost the same type of temperature dependence as the dhcp form, but has a 10% lower magnitude.

I. INTRODUCTION

The electrical-resistivity measurements reported here on high-purity α -La [double hexagonal close packed (dhcp)] were undertaken so that the lattice resistivity and magnetic resistivity components of β -Ce (dhcp) could be separated and analyzed.¹⁻³ The lattice resistivities of α -La and β -Ce were assumed to be the same. In addition we have found high-purity La invaluable in studies of the superconductivity of dhcp La alloys.^{4,5}

La is a trivalent metal with the three conduction electrons in the $5d6s$ configuration. Since it has no $4f$ electrons it is essentially nonmagnetic. Previous measurements on the electrical resistivity of La have been reported by James *et al.*,⁶ Alstad *et al.*,⁷ and Krizek.⁸ Alstad *et al.*⁷ reported that each time the originally fcc sample was cycled to helium temperature it contained more dhcp phase. This is in keeping with our recent metallurgical observations that pure La can be obtained and retained at room temperature only in the dhcp phase. Interstitial impurities stabilize the fcc form.

II. SAMPLE PREPARATION

Two separate batches of La were used to prepare the dhcp samples. The analyses are given in Table I. The two samples, A and B from batch 1 were cut from a single piece of metal which had been treated in the following manner: a rectangular parallelepiped $5 \times 9 \times 18$ mm was cut out of a large as-vacuum-melted billet with a jeweler's saw. The cold worked surface introduced by sawing was removed by electropolishing in a perchloric-methanol bath.⁹ The sample was sheathed in tantalum and reduced 25% in thickness by rolling at room temperature. The sample was heated to 440 °C for 4 h., cooled to 150 °C, then reheated to 260 °C for 1 week in a 1×10^{-8} Torr (1.3×10^{-6} Pa) vacuum. A vacuum fusion analysis for H, O, and N after the heat

treatment showed no contamination had occurred. Debye-Scherrer x-ray patterns of two needle-shaped samples prepared as described earlier^{9,10} showed only lines of the dhcp form, which indicates that the samples contain no more than 2% fcc La (the lower limit for detecting β -La). Metallographic examination of a piece adjacent to the resistivity samples showed randomly oriented grains with grain dimensions ranging from 4 to 100 μ m on an edge. Two resistivity samples each about 1.5 cm \times 2 cm were cut out of the heat-treated piece with a jeweler's saw. The sides were ground parallel and the sample was electropolished to remove the cold worked surface.

The two samples C and D from batch 2 were cut with a low-speed diamond saw from a large (2 kg)

TABLE I. Analyses^a of La metal in atomic ppm. (Weight ppm in parentheses.)

Impurity	Batch 1 (A and B)	Batch 2 (C and D)	fcc sample
H	412 (3)	275 (2)	1788 (13)
C	276 (24)	93 (8)	150 (13)
N	218 (22)	20 (2)	2083 (210)
O	330 (38)	252 (29)	1996 (230)
F	190 (26)	511 (70)	1857 (254)
Fe	6 (2.6)	6 (2.6)	48 (19)
Ni	5	22	13
Cu	2	7	N.D. ^b
Ta	20	3	N.D.
Y	3	3	N.D.
Ce	20	4	N.D.
Gd	2	1	2000
Ho	2	0.2	N.D.

^aAll naturally occurring metallic impurities were determined by mass spectrometry (those not listed were present in less than 1 ppm); O, N, and H by vacuum fusion; C by a combustion-chromatography method; and F by the formation, distillation, and determination of fluosilicic acid method.

^bN.D.: not determined.

billet which had been melted in a Ta crucible and cooled slowly. Sample *C* was cut parallel to a tangent of the cylindrical billet and then *D* was cut perpendicular to the tangent and to the radius. Metallographic examination did not indicate preferred orientation.

The fcc sample was prepared from a La-0.2-at.-%-Gd alloy of low purity (see Table I). As discussed below it is difficult to obtain the fcc form using pure La; it was by chance that the fcc sample which was available to us had Gd in it.

III. RESULTS AND DISCUSSION

The electrical resistivity of four different specimens of dhcp La and of one specimen of fcc La-Gd alloy were measured from 5.2 to 310 K. The residual resistivity, room temperature resistivity, resistivity ratio, and superconducting ordering temperature T_c , determined from resistivity measurements and by a mutual inductance technique for each sample are shown in Table II. The $\rho_{5.2\text{ K}}$ data of the table are normal-state values extrapolated from above 6.3 K. It may be seen in the table that there is a spread of 1.1 out of 62 $\mu\Omega\text{ cm}$ in the room-temperature resistivities of the dhcp samples.

Since samples *C* and *D* were cut orthogonal to each other from the same ingot we conclude that the small crystallites had random orientations. With the residuals subtracted the resistivity data for all dhcp samples fell within a 2% range. Sample *B* had the lowest residual resistivity and the lowest room-temperature resistivity. The results for this sample are shown in Fig. 1. The insert of the figure shows the raw low-temperature data. Since resistivity measurements exaggerate the amount of the higher- T_c component by a shorting-out process, the small resistivity change at 6.1 K (T_c for the fcc allotrope) shows that very little fcc La is present. The ac susceptibility, a more reliable indicator with about 1% detectability limit, showed that samples *A* and *B* were 100% dhcp and that all sample superconducting transitions were narrow with widths about 0.03 K (10% to 90% superconducting). This gave us assurance of high sam-

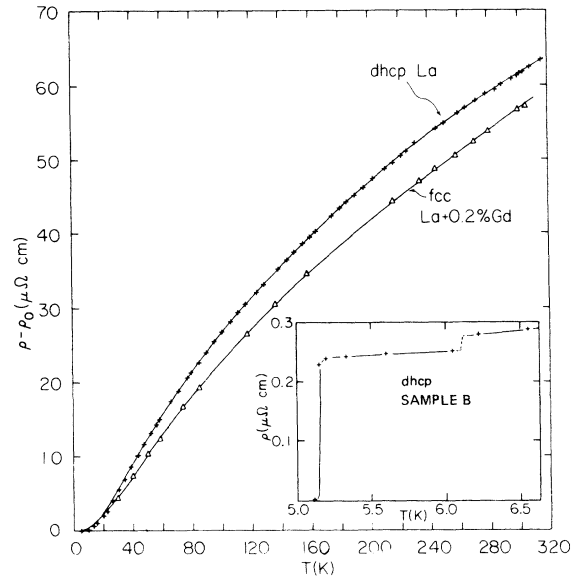


FIG. 1. Electrical resistivity vs temperature for α -La (dhcp) and for β -La (fcc)+0.2-at.-% Gd. The dhcp data are for sample B. The curves are guides for the eye.

ple quality. The raw data for sample *B* are available.¹¹

Many attempts were made to retain the fcc form at room temperature of the chemically pure La which had been used for the dhcp studies, but each failed. Thus we decided to use a fcc sample of La (containing 0.2-at.-% Gd) which had been prepared 12 years ago¹² and had been kept sealed in helium since that time. A Debye-Scherrer pattern of a solid sample prepared as described earlier^{9,10} showed the sample remained in the fcc form. It was in excellent condition and had a residual of only 1.3 $\mu\Omega\text{ cm}$, most of which we attribute to the interstitial and Gd impurities. The results of resistivity measurements for this sample are also shown in Fig. 1. Since this sample is cubic no specimen orientation problem could be involved. One must bear in mind that there is a total of 0.79-at.-% interstitial impurities in this sample. Except at very low temperatures it is unlikely that

TABLE II. La sample properties.

Sample	$\rho_{5.2\text{ K}}$ ($\mu\Omega\text{ cm}$)	$\rho_{300\text{ K}} - \rho_{5.2\text{ K}}$ ($\mu\Omega\text{ cm}$)	$\frac{\rho_{300\text{ K}} - \rho_{5.2\text{ K}}}{\rho_{5.2\text{ K}}}$	$T_c(\rho)$ (K)	$T_c(\chi)$ (K)
A dhcp	0.43	62.4	145	5.17	5.12
B dhcp	0.27	61.3	227	5.15	5.12
C dhcp	0.43	62.2	144	5.22	5.12
D dhcp	0.50	62.0	124	5.21	5.12
E fcc	1.3	56.6	44	5.46	5.50

the Gd impurity has a significant effect on the electrical-resistivity results. In addition to its contribution to the residual resistivity, the Gd impurity lowers the superconducting transition temperature by ~ 0.8 K,^{5,12} which is consistent with the 0.6-K lowering observed here.

A power-law analysis of the low-temperature data was not possible because of the superconducting transition. However, the continuous concave downward curvature and lack of linearity of the data near 300 K is significant and is in keeping with recent observations by Fisk and Webb¹³ about high-temperature electrical resistivity of superconductors.

It will be interesting to see what results might

be forthcoming for single-crystal dhcp La. The procedure for obtaining such samples is not known; nevertheless, efforts to grow them continue. It is estimated that the average room-temperature single-crystal resistivity will be within 2% of the results reported here.

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