行政院國家科學委員會專題研究計畫成果報告

$(R_{1-x-z}Ca_xPr_z)Ba_{1.5}Sr_{0.5}Cu_3O_y$ 超導系列 T_c 與組成的關係

Correlation of Tc with compositions in the $(R_{1-x-z}Ca_xPr_z)Ba_{1.5}Sr_{0.5}Cu_3O_y$ superconducting series

關鍵詞：$(R_{1-x-z}Ca_xPr_z)Ba_{1.5}Sr_{0.5}Cu_3O_y$、超導性、晶胞參數、電洞濃度、氧含量。

1. 摘要

　　本研究以固態反應法製備數系列具有四方晶系三層鈦酸鈣結構且完全氧化的單相化合物，其化學組成為 $(La_{1-x-z}Ca_xPr_z)Ba_{1.5}Sr_{0.5}Cu_3O$. 在 Pr 系列中，當 z = 0 時，$(La_{1-z}Pr_z)Ba_{1.5}Sr_{0.5}Cu_3O_y$ 的 T_c 為 54 K；z 增加時，T_c 快速下降；當 z = 0.30 時，變為絕緣體. 在 Ca 系列中，當 x = 0 時，$(La_{1-x}Ca_x)Ba_{1.5}Sr_{0.5}Cu_3O_y$ 的 T_c 為 54 K；x 增加時，T_c 上升；當 x = 0.20 時，T_c 變為 73 K . 0.20 是 Ca 離子在此系列化合物中的溶解度極限. 當 Pr 的含量固定為 0.10 、 0.20 和 0.30 時，$(La_{1-x-z}Ca_xPr_z)Ba_{1.5}Sr_{0.5}Cu_3O_y$ 的 T_c 隨著 Ca 離子取代量增加而增加. 假設樣品中 Pr 離子有 20% 為 Pr^{4+} 時，計算所得的 p_{sh} (CuO_2 面上的電洞濃度) 與 T_c 相關. 很明顯地，將 Ca 離子放在 La 位置 (單位晶胞的中心位置) 上，可以增加超導轉變溫度，它也有增加 CuO_2 面上電洞濃度的效應. 同理，Pr^{4+} 離子在 La 位置上有電洞充填的效應. p_{sh} 與 ΔOF 呈線性關係，ΔOF 等於 Ca 離子填在 La 位置的佔有率減掉 Pr^{4+} 離子填在 La 位置的佔有率 ($\Delta OF = OF_{Ca/La} - OF_{Pr(IV)/La}$). 本研究進一步確認 Ca 和 Pr^{4+} 離子對 CuO_2 面上電洞濃度的影響. 含有相同 p_{sh} 的樣品隨著 Pr 含量增加，T_c 繼續下降，推測 Pr 在 La 位置上還有其他破壞超導性的效應.

Keywords: $(R_{1-x-z}Ca_xPr_z)Ba_{1.5}Sr_{0.5}Cu_3O_y$, superconductivity, cell parameters, hole concentration, oxygen stoichiometry.

1. Abstract

　　Several series of fully oxygenated single phase $(La_{1-x-z}Ca_xPr_z)Ba_{1.5}Sr_{0.5}Cu_3O_y$ compounds with tetragonal triple-perovskite structure were prepared by a solid state reaction method. In the Pr series, T_c of $(La_{1-z}Pr_z)Ba_{1.5}Sr_{0.5}Cu_3O_y$ is quickly dropped from 54 K for z = 0 to insulator for z greater than 0.30. However, in the Ca series, T_c of $(La_{1-x}Ca_x)Ba_{1.5}Sr_{0.5}Cu_3O_y$ is increased from 54 K for x = 0 to 73 K for x = 0.20. 0.20 is the solubility limit of the Ca ion in this series. Keeping the content of Pr as a constant, when z = 0.10, 0.20 and 0.30, T_c of $(La_{1-x-z}Ca_xPr_z)Ba_{1.5}Sr_{0.5}Cu_3O_y$ is also increased by increasing the amount of Ca ion in each respective series. Assuming 20 % of Pr ion is in the tetravalent state, p_{sh} (the hole concentration in the CuO_2 layer) for all the compounds is calculated and it is correlated to T_c. Apparently, introducing Ca ion into La site (the center position of the unit-cell), T_c is increased and the hole concentration in the CuO_2 layer is also increased. On the other hand, introducing Pr^{4+} ion into La site, hole concentration in the CuO_2 layer is decreased. A linear relationship is found in between p_{sh} and ΔOF ($\Delta OF = OF_{Ca/La} - OF_{Pr(IV)/La}$), where $OF_{Ca/La}$ is the occupancy factor of the Ca ion in the La site and $OF_{Pr(IV)/La}$ is the occupancy factor of the Pr(IV) ion in the La site. The hole doping and filling effects caused by the Ca and Pr^{4+} ions, respectively is further demonstrated. With a constant p_{sh}, T_c is decreased with increasing the amount of Pr

content, indicating a further deterioration of the superconductivity by Pr ion in the La site.

2. INTRODUCTION

Soon after the discovery of the 90 K $YBa_2Cu_3O_{7-\delta}$ superconductor (1), a series of 90 K $RBa_2Cu_3O_y$ (R = rare-earth, except Ce, Pr and Tb) superconductors is found (2,3). All of the 90 K $RBa_2Cu_3O_y$ superconductors have orthorhombic triple-perovskite structure (4,5), indicating that both T_c and the structure of $RBa_2Cu_3O_y$ are insensitive to the substitution of the R ion.

On the other hand, if Ba is partially substituted by Sr ion in the $R(Ba_{2-x}Sr_x)Cu_3O_{7-\delta}$ series, T_c decreases with increasing the amount of Sr ion and the decreasing rate of the T_c is dependent on the ionic radius of the R ion (6,7). For example, T_c of $YBa_{2-x}Sr_xCu_3O_y$ decreases from 92 to 83 K when x increases from 0 to 1.00 (6). T_c of $LaBa_{2-x}Sr_xCu_3O_y$ changes from 90 to 50 K when x increases from 0 to 0.80. The same phenomenon is also found in the $(R_{1-x}Pr_x)Ba_2Cu_3O_{7-\delta}$ system (8-10).

It is well known that substitution part of a divalent for a trivalent cation, it has the hole doping effect on the CuO_2 plane and T_c is increased in the $La_{2-x}A_xCuO_4$ system with A = Ca, Sr or Ba (11-13).

In a series study in the $(La_{1.5-x}Ca_x)(Ba_{1.5-z}Ca_z)Cu_3O_y$ system, Ca is found in the R site (the center position of a triple-perovskite unit-cell) if the amount of Ca is less than 0.4. It has a hole doping effect on the CuO_2 layer. T_c increases with increasing the amount of Ca ion in the samples (14-16). It is also found that introducing La ion into Ba site, hole concentration of the CuO_2 layer decreases with increasing the amount of La ion (17).

In the fully oxygenated $YBa_2Cu_3O_y$ compound, partly substitution of the Y by Ca ion, T_c drops quickly to 50 K with the amount of Ca equals to 0.20 (18). It is not possible to find the hole doping effect for Ca in Y site. However, in an oxygen deficient sample, T_c of $YBa_2Cu_3O_y$ is lower than that of $Y_{0.8}Ca_{0.2}Ba_2Cu_3O_y$ (19). The hole doping effect of Ca is thus appeared.

In this report, a series of fully oxygenated samples with the compositions as $(La_{1-x-z}Ca_xPr_z)Ba_{1.5}Sr_{0.5}Cu_3O_y$ were prepared in order to find the hole doping effect caused by the Ca ion and the hole filling effect caused by the existence of the Pr^{4+} ion. The correlation of the T_c to the hole concentration in the CuO_2 plane and to the occupancy factors of the various valent cations in the R site is also investigated.

3. EXPERIMENTAL

Solid state reaction method was employed for making $(La_{1-x-z}Ca_xPr_z)Ba_{1.5}Sr_{0.5}Cu_3O_y$ compounds with $0 \leq x \leq 0.20$, $0 \leq z \leq 1.00$. La_2O_3 and Pr_6O_{11} was preheated at 1000°C for 10 h and kept in desiccator prior to use. Stoichiometric amounts of La_2O_3, Pr_6O_{11}, $BaCO_3$, $SrCO_3$ and CuO were weighed, ground thoroughly with a Retsch Spectro Mill Type MS for 30 min. and then ground further manually. Absolute alcohol was added to enhance the grinding effect. Mixed powder was calcined at 950°C for 18 h in a box furnace with three intermittent grindings. It was reground and pressed into half inch diameter pellet. For the sample without Ca ion, the resultant pellet was sintered at 950°C for 16 h in a tube furnace with static air and then cooled under a rate of 100°C/h to 400°C in the flowing nitrogen atmosphere. It was annealed at 400°C in the flowing oxygen atmosphere for 12 h and followed by furnace cooling to room temperature. For the sample containing Ca ion, the pellet was sintered at 930°C in the flowing nitrogen atmosphere for 12 h, quenched to room temperature, then annealed at 400°C furnace in the flowing oxygen atmosphere for 12 h and followed by furnace cooling to room temperature.

Purity of the sample was examined by a Mac Science MXP3 X-ray powder diffractometer equipped with a Cu K_α radiation, Ni-filter and graphite

monochromater. Peaks of the X-ray diffraction (XRD) pattern were corrected with $K_{\alpha 2}$ elimination and calibrated with an internal Si standard. Unit-cell parameters were calculated using least squares refinement. T_c was measured by a SQUID magnetometer. Oxygen stoichiometry was obtained by iodometric titration according to the report by Appelman et al. (20). 20 % of the Pr ion was assumed to be in the Pr^{4+} oxidation state according to our previous experience (21). p_{sh} of the samples in the CuO_2 layers was calculated according to the report by Tokura et al. (22).

4. RESULTS AND DISCUSSION

All the samples prepared in this report are supposed to be fully oxygenated. In the Pr series, all the samples with $0 \leq z \leq 1.00$, $(La_{1-z}Pr_z)Ba_{1.5}Sr_{0.5}Cu_3O_y$ have tetragonal triple- perovskite crystal structure. In the Ca series, the allowed solubility limit for Ca ion in the $(La_{1-x}Ca_x)Ba_{1.5}Sr_{0.5}Cu_3O_y$ series is found as $0 \leq x \leq 0.20$. It has tetragonal triple- perovskite structure too. Table 1 shows the unit-cell parameters, a and c axes and V for all of the samples prepared. Both Pr and Ca ions are smaller than the La ion, a and c axes and V of the $(La_{1-x-z}Ca_xPr_z)Ba_{1.5}Sr_{0.5}Cu_3O_y$ compounds are decreased monotonically with the increase of the amount of substitution.

T_c of the Pr series is quickly decreased from $z = 0$ at 54 K to $z = 0.20$ at 24 K. For $z = 0.30$, it becomes insulator. Results are listed in Table 2.

Introducing Sr into $LaBa_2Cu_3O_y$, T_c of $LaBa_{1.5}Sr_{0.5}Cu_3O_y$ is 54 K listed in Table 2. Partly substitution of La by Ca ion, T_c was increased from 54 to 73 K for $0 \leq x \leq 0.20$ in the $(La_{1-x}Ca_x)Ba_{1.5}Sr_{0.5}Cu_3O_y$ series. Introducing Ca ion into La site (the center position of the unit-cell), it enhances the superconductivity of the $LaBa_{1.5}Sr_{0.5}Cu_3O_y$ system.

Fixing the amount of Pr to 0.10, T_c of $(La_{1-x}Ca_xPr_{0.1})Ba_{1.5}Sr_{0.5}Cu_3O_y$ series is also increased with increasing x. The same

phenomena are also found in fixing the amount of Pr to 0.20 and 0.30 in the fully oxygenated $(La_{1-x-z}Ca_xPr_z)Ba_{1.5}Sr_{0.5}Cu_3O_y$ compounds. Figure 1 shows the T_c versus Ca content for Pr = 0 - 0.30 samples. T_c drops due to existence of the Pr ion and T_c increases by the addition of Ca ion.

Plotting T_c versus p (average hole concetration in each copper oxygen plane, assuming all the Pr ion is in the trivalent state), no correlation is found between these two parameters.

In our previous study, we found that Pr ion in the fully oxygenated samples contains about 20 % of Pr^{4+} ion (21). If we assumed that 20% of the Pr ion is in the Pr^{4+} state, the corrected average hole concentration (p*) is also plotted with T_c shown in Figure 2. No correlation is also observed. We know that there are two copper sites, Cu(1) and Cu(2) in a triple-perovskite unit-cell (4), only the hole concentration in the CuO_2 layers (p_{sh}) is responsible for the superconductivity in 123 compounds (4). We should separate the hole concentration in the $Cu(2)O_2$ layers (p_{sh}) from those in the Cu(1)O layer (p_{ch}). In order to find p_{sh}, Tokura method (22) is employed for the calculation. Plotting T_c versus p_{sh} in Figure 4, four curves corresponding to different amount of Pr ion are found. T_c is correlated to p_{sh}. However, with the same amount of p_{sh}, samples containing higher concentration of Pr ion has a lower T_c, indicating that superconductivity is further influenced by the existence of the Pr ion, such as, pair breaking or the hybridization of the Pr 4f electrons with the O 2p atom in the CuO_2 layer (8-10).

In the introduction, we mentioned about the hole doping effect caused by the substitution of the divalent to the trivalent cation and hole filling effect caused by the substitution of the tetravalent to the trivalent cation. The net effect of the hole doping on the CuO_2 plane may be correlated to the difference of the occupancy factors ($\Delta OF = OF_{Ca/La} - OF_{Pr(IV)/La}$), where $OF_{Ca/La}$ is the occupancy factor of the Ca ion in the La site and $OF_{Pr(IV)/La}$ is the occupancy factor of the Pr(IV) ion in the La site. Plotting ΔOF

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versus p_{sh}, a straight line is observed with $\Delta OF = 3.57\, p_{sh} - 0.69$, the fitness number is 0.98 for all the samples. It is shown that p_{sh} calculated by Tokura method is directly correlated to ΔOF. The idea of calculating p_{sh} given by Tokura et al. is based on the difference of the occupancy factors. The same result is also observed in the $(La_{1.5-x}Ca_x)(Ba_{1.5-z}Ca_z)Cu_3O_y$ system (17).

In conclusion, we proved that (1) Ca is a hole dopant if it is introduced into the center position of the triple-perovskite unit-cell and Pr^{4+} is a hole filler in the same position. (2) The more the Pr ion in the La site, the more the superconductivity is deteriorated if the p_{sh} is kept as a constant. T_c is further destroyed by Pr ion.

ACKNOWLEDGMENT

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表 1　$La_{1-x-z}Ca_xPr_zBa_{1.5}Sr_{0.5}Cu_3O_y$ 系列化合物晶格參數

	a (Å)	c (Å)	V (Å^3)
$La_{1-z}Pr_zBa_{1.5}Sr_{0.5}Cu_3O_y$			
z = 0	3.9047(8)	11.758(6)	179.27(7)
0.10	3.9042(8)	11.716(6)	178.58(9)
0.20	3.9038(5)	11.714(3)	178.53(5)
0.30	3.9038(5)	11.695(4)	178.23(9)
0.40	3.8990(6)	11.700(2)	177.86(5)
0.50	3.8979(7)	11.673(3)	177.36(7)
0.60	3.8888(4)	11.666(2)	176.42(4)
0.70	3.8869(7)	11.670(2)	176.32(6)
0.80	3.8857(4)	11.650(2)	175.90(4)
0.90	3.8841(7)	11.649(3)	175.75(6)
1.00	3.8825(4)	11.650(2)	175.62(4)
$La_{1-x}Ca_xBa_{1.5}Sr_{0.5}Cu_3O_y$			
x = 0	3.9047(8)	11.758(6)	179.27(7)
0.05	3.8987(7)	11.763(2)	178.80(6)
0.10	3.8979(8)	11.742(3)	178.40(7)
0.15	3.8969(9)	11.729(3)	178.12(7)
0.20	3.8966(9)	11.686(4)	177.44(8)
$La_{0.9-x}Ca_xPr_{0.1}Ba_{1.5}Sr_{0.5}Cu_3O_y$			
x = 0	3.9042(8)	11.716(6)	178.58(9)
0.05	3.8971(3)	11.789(5)	179.90(9)
0.10	3.9054(5)	11.723(3)	178.80(6)
0.15	3.9045(8)	11.718(2)	178.64(4)
0.20	3.9036(3)	11.708(1)	178.42(3)
$La_{0.8-x}Ca_xPr_{0.2}Ba_{1.5}Sr_{0.5}Cu_3O_y$			
x = 0	3.9038(5)	11.714(3)	178.53(5)
0.05	3.8971(3)	11.698(1)	177.67(3)
0.10	3.8962(4)	11.688(2)	177.44(4)
0.15	3.8905(8)	11.677(3)	176.75(7)
0.20	3.8885(4)	11.665(2)	176.39(4)
$La_{0.7-x}Ca_xPr_{0.3}Ba_{1.5}Sr_{0.5}Cu_3O_y$			
x = 0	3.9038(5)	11.695(4)	178.23(9)
0.05	3.8941(5)	11.690(2)	177.28(5)
0.10	3.8927(7)	11.679(2)	176.98(6)
0.15	3.8907(3)	11.675(1)	176.75(3)
0.20	3.8838(6)	11.660(2)	175.88(5)

表 2 $La_{1.0-x-z}Ca_xPr_zBa_{1.5}Sr_{0.5}Cu_3O_y$ 系列化合物的超導轉變溫度、氧含量、電洞濃度和佔有率差

	T_c	y	p	p^*	p_{sh}	ΔOF
$La_{1-z}Pr_zBa_{1.5}Sr_{0.5}Cu_3O_y$						
$z = 0$	54	6.902(4)	0.268(3)	0.268	0.201	0
0.10	44	6.904(2)	0.269(1)	0.263	0.192	- 0.02
0.20	24	6.906(4)	0.271(2)	0.257	0.183	- 0.04
0.30	na	6.909(4)	0.273(2)	0.253	0.175	- 0.06
0.40	na	6.910(3)	0.273(2)	0.247	0.165	- 0.08
0.50	na	6.916(2)	0.277(2)	0.244	0.158	- 0.10
0.60	na	6.941(4)	0.294(2)	0.254	0.161	- 0.12
0.70	na	6.945(4)	0.297(3)	0.250	0.153	- 0.14
0.80	na	6.974(1)	0.316(1)	0.263	0.157	- 0.16
0.90	na	6.980(4)	0.320(2)	0.260	0.150	-0.18
1.00	na	6.982(5)	0.321(3)	0.255	0.141	- 0.20
$La_{1-x}Ca_xBa_{1.5}Sr_{0.5}Cu_3O_y$						
$x = 0$	54	6.902(4)	0.268(3)	0.268	0.201	0
0.05	66	6.878(3)	0.269(2)	0.269	0.214	0.05
0.10	68	6.847(3)	0.264(2)	0.264	0.224	0.10
0.15	71	6.819(3)	0.263(2)	0.263	0.235	0.15
0.20	73	6.794(3)	0.262(2)	0.262	0.247	0.20
$La_{0.9-x}Ca_xPr_{0.1}Ba_{1.5}Sr_{0.5}Cu_3O_y$						
$x = 0$	44	6.904(2)	0.269(1)	0.263	0.192	- 0.02
0.05	50	6.865(2)	0.260(2)	0.253	0.198	0.03
0.10	56	6.856(2)	0.271(1)	0.264	0.218	0.08
0.15	62	6.845(4)	0.280(3)	0.273	0.238	0.13
0.20	63	6.809(3)	0.273(2)	0.266	0.245	0.18
$La_{0.8-x}Ca_xPr_{0.2}Ba_{1.5}Sr_{0.5}Cu_3O_y$						
$x = 0$	24	6.906(4)	0.271(2)	0.257	0.183	- 0.04
0.05	33	6.886(3)	0.274(2)	0.261	0.198	0.01
0.10	40	6.862(5)	0.275(3)	0.261	0.211	0.06
0.15	45	6.840(2)	0.277(1)	0.263	0.225	0.11
0.20	47	6.809(3)	0.273(2)	0.259	0.235	0.16
$La_{0.7-x}Ca_xPr_{0.3}Ba_{1.5}Sr_{0.5}Cu_3O_y$						
$x = 0$	na	6.909(4)	0.273(2)	0.253	0.175	- 0.06
0.05	14	6.896(3)	0.281(2)	0.261	0.193	- 0.01
0.10	26	6.872(2)	0.281(2)	0.261	0.206	0.04
0.15	27	6.851(4)	0.284(3)	0.264	0.221	0.09
0.20	34	6.829(3)	0.286(2)	0.266	0.235	0.14

Assume 20% Pr^{4+}

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圖 1. La$_{1-x-z}$Ca$_x$Pr$_z$Ba$_{1.5}$Sr$_{0.5}$Cu$_3$O$_y$ 系列化合物的超導臨界溫度與 Ca 取代量的關係圖

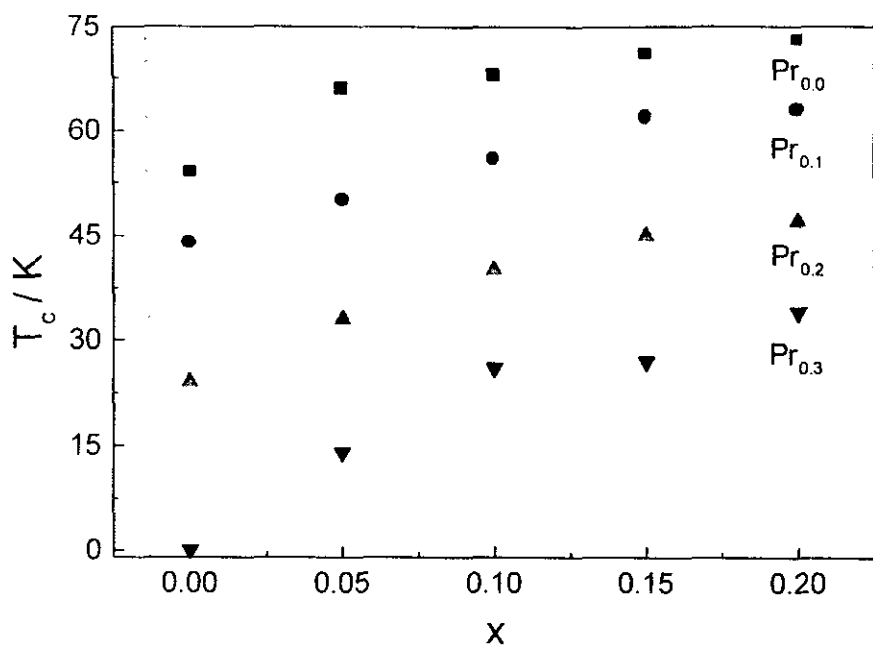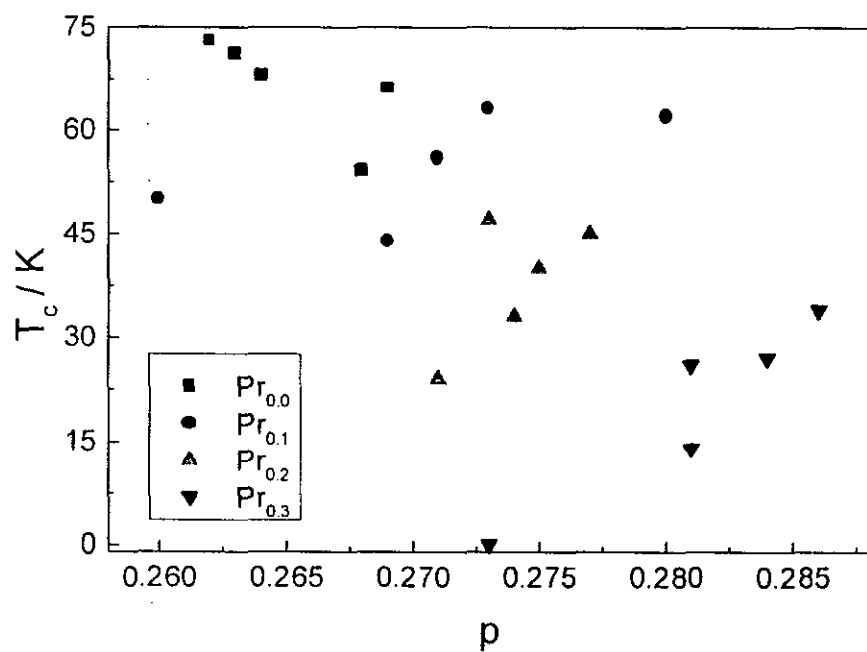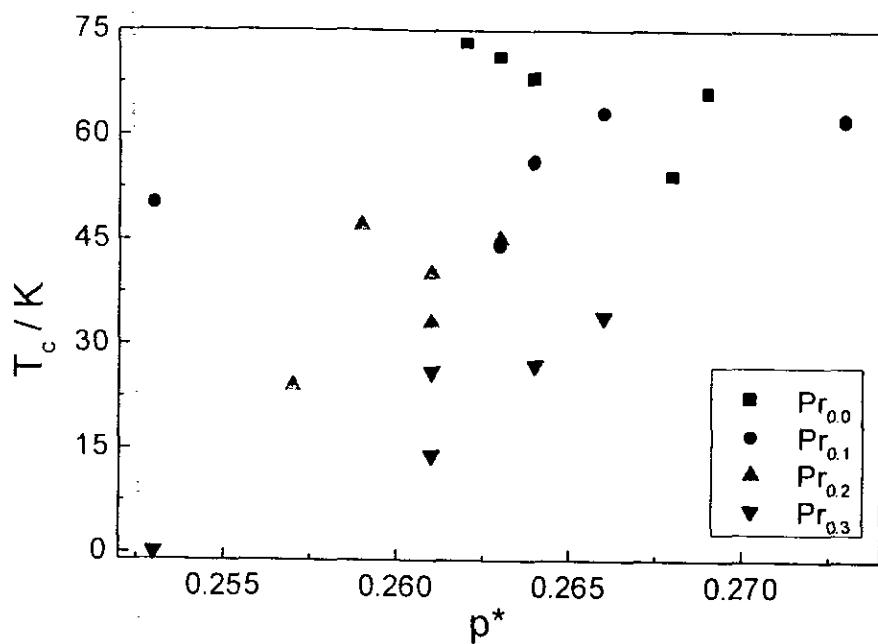圖 2. La$_{1-x-z}$Ca$_x$Pr$_z$Ba$_{1.5}$Sr$_{0.5}$Cu$_3$O$_y$ 系列化合物的超導臨界溫度與電洞濃度(p)的關係圖

圖 3. $La_{1-x-z}Ca_xPr_zBa_{1.5}Sr_{0.5}Cu_3O_y$ 系列化合物的超導臨界溫度與校正後的電洞濃度(p^*)的
關係圖

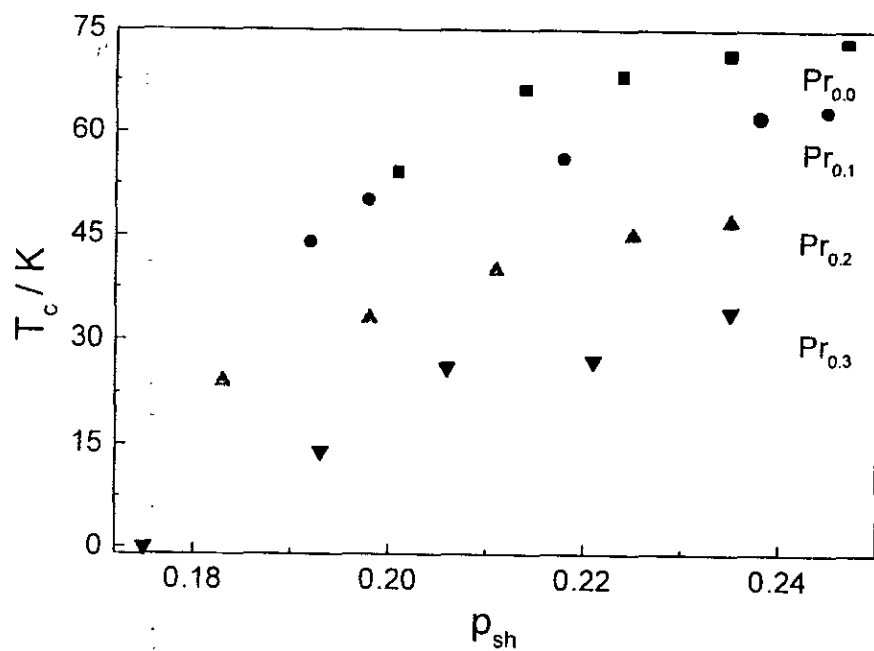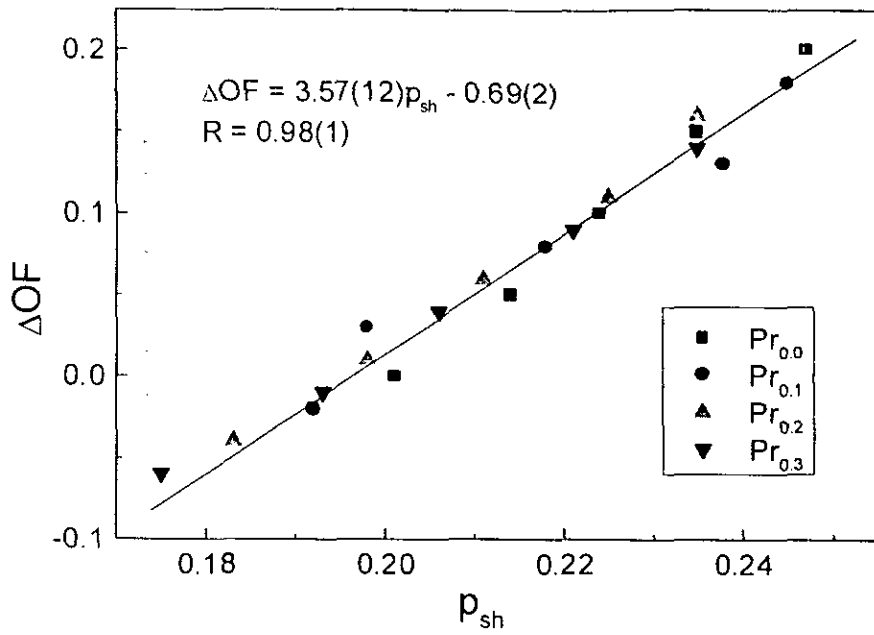圖 4. $La_{1-x-z}Ca_xPr_zBa_{1.5}Sr_{0.5}Cu_3O_y$ 系列化合物的超導臨界溫度與電洞濃度(p_{sh})的關係圖

$$\Delta OF = 3.57(12)p_{sh} - 0.69(2)$$
$$R = 0.98(1)$$

圖 5. $La_{1-x-z}Ca_xPr_zBa_{1.5}Sr_{0.5}Cu_3O_y$ 系列化合物的 ΔOF 與電洞濃度(p_{sh})的關係圖