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REACTIONS BETWEEN $YBa_2Cu_3O_{7-\delta}$ AND La_2O_3 AND $SrCO_3$

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Abstract

Solid state reactions at 925°C between the high-T_c ceramic superconductor $YBa_2Cu_3O_{7-\delta}$ and La_2O_3 and $SrCO_3$, respectively, mixed in various molar ratios $r=MeO_n/YBa_2Cu_3O_{7-\delta}$, were studied using X-ray powder diffraction and scanning electron microscopy. The reaction between $YBa_2Cu_3O_{7-\delta}$ and La_2O_3 yielded $(La_{1-x}Ba_x)_2CuO_{4-\delta}$, with $x\approx0.075-0.10$, $La_{2-x}Ba_{1+x}Cu_2O_{6-\delta}$, with $x\approx0.2-0.25$ and La-doped $(Y_{1-x}La_x)_2BaCuO_5$, with $x\approx0.10-0.15$. For $r=3.0$, Y-doped La_2BaCuO_5 resulted also. The reaction between $YBa_2Cu_3O_{7-\delta}$ and $SrCO_3$ yielded $(Sr_{1-z}Ba_z)_2CuO_3$, with $z\approx0.1$, $Y_2(Ba_{1-z}Sr_z)CuO_5$, with $z=0.1-0.15$, and a nonsuperconducting compound with an approximate composition of $Y(Ba_{0.5}Sr_{0.5})_5Cu_{3.5}O_{10\pm\delta}$. At values of $r\leq2.0$, unsubstituted $YBa_2Cu_3O_{7-delta}$ was found in the reaction products.

Keywords: La_2O_3, $SrCO_3$, $YBa_2Cu_3O_{7-\delta}$, superconductors

Introduction

Isomorphic substitutions for host ions in the ceramic superconductor $YBa_2Cu_3O_{7-\delta}$ (labeled below as YBCO or Y_{123}) are possible for a great variety of ions which have ionic radii and oxidation states close to those of the host ions.

In a couple of previous papers solid state reactions between YBCO and oxides of potential substituents for yttrium, such as Eu, Ca, Th, Sc and Ce – [1] – and for copper, such as Fe, Co and Zn [2] were studied. It was concluded that the existence of substituted Y_{123}-phases seems to largely depend on chemical stability relations between the substituted YBCO and compounds of the oxide of the particular substituent ion with oxides of Ba, Y and Cu.

The purpose of this work is to study the solid state reactions between $YBa_2Cu_3O_{7-\delta}$ and compounds of elements which have been reported to substitute at the Ba-site, *i.e.* cations such as La^{3+}(with ionic radius $R_X=1.28$Å) and Sr^{2+} ($R_X=1.32$Å). Given the differences in ionic radii between La^{3+}, Sr^{2+} and

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Ba^{2+} (R_X=1.52Å) the main effect of the La and Sr-substitution for Ba is a shortening of the lattice parameters, especially the c-axis [3, 4].

La-substituted $Y(Ba_{1-x}La_x)_2Cu_3O_{7+\delta}$ was reported to have orthorhombic structure for values $x<0.15$ and tetragonal structure for values of $0.15<x\leq0.36$ [3, 5]. These solid solutions are superconducting, with reduced T_c in the tetragonal state too.

Sr can replace Ba in $Y(Ba_{1-z}Sr_z)_2Cu_3O_{7-\delta}$ up to $z=0.4$. A linear depression of the T_c in combination with stabilization of the tetragonal crystalline phase with increasing Sr concentration has been reported. For $z>0.4$ impurity phases, such as $Y_2(Ba_{0.9}Sr_{0.1})_2CuO_5$ and $(Sr_{2/3}Ba_{1/3})_{14}Cu_{24}O_{41}$ emerge [6, 7].

The phase analysis of the reaction products requires a thorough knowledge about the compounds in the investigated systems. The Y–Ba–Cu–O system exhibits three quaternary phases at 925–950°C, in air or oxygen: the black, high-T_c superconductor $YBa_2Cu_3O_{7-\delta}$ with orthorhombic structure (Y_{123}) [8], the green Y_2BaCuO_5 with orthorhombic structure (Y_{211}) [9] and one barium-rich compound with approximate composition $YBa_3Cu_2O_x$ (Y_{132}), $YBa_4Cu_3O_{8.5+\delta}$ (Y_{143}) or $Y_{1.0-1.33}Ba_8Cu_4O_{13.5\pm\delta}$ (Y_{184}), having cubic or tetragonal structures [10–17].

In addition to the 30K-superconductor $(La_{1-x}Bax)2CuO_{4-\delta}$, with $x\approx0.075$ [18–20] and 90K-superconductor $La_{3-x}Ba_{3+x}Cu_6O_{14\pm\delta}$, with $0.0\leq x\leq0.8$ [20–22], there have been reported three other quaternary phases in the La–Ba–Cu–O system at 925–950°C, i.e. $La_4BaCu_5O_{13+\delta}$, labeled La_{415} [20, 23, 24], $La_{4-2x}Ba_{2-2x}Cu_{2-x}O_{10-2x}$, with $0.0\leq x\leq0.25$ (La_{211}) [20, 25, 26] and $La_{2-x}Ba_{1+x}Cu_2O_{6-x/2}$, ($La_{212}$) with $0.2\leq x\leq0.8$ [7, 20, 26, 27].

Solid solutions of the type $(Sr,Y,Ba)_{14}Cu_{24}O_{41}$ and $Y_2(Ba,Sr)CuO_5$ were reported to exist in the Y–Ba–Sr–Cu–O system [28]. However, no quaternary compounds were reported in the Y–Sr–Cu–O system [29].

Experimental

Solid state reactions between $YBa_2Cu_3O_{7-\delta}$ (the coefficient δ in this article is only a qualitative indication of the oxygen nonstoichiometry of the oxide compounds) prepared by standard ceramic method, and La_2O_3 and $SrCO_3$, respectively, both of p.a. quality (Merck), were performed by heat treatment of tablets of well homogenized powdered mixtures, for values of the molar ratio $r=MeO_n/YBCO=0.1$, 0.2, 0.4, 1.0, 2.0 and 3.0, respectively, at 925±10°C, in air, for 18–24 h in an electric furnace. The samples were allowed to cool slowly to room temperature, reground and subjected to a second heat treatment in flowing oxygen at the same temperature for 1 h, followed by heat treatment in oxygen at 450°C for 24 h.

In connection with the phase analysis of the reaction products, solid solutions were prepared: $La_{4-2x}Ba_{2-2x}Cu_{1-x}O_{10-x}$, with $x=0.0$ and 0.20, $(La_{1-x}Ba_x)_2CuO_{4-\delta}$ with $x=0.075$ and 0.10, $La_{2-x}Ba_{1+x}Cu_2O_{6-\delta}$, with $x=0.2$, 0.3, 0.5 and 0.7, La- and Sr-substituted Y_2BaCuO_5, Ba-substituted Sr_2CuO_3 and a compound with chemical composition $Y(Sr_{0.5}Ba_{0.5})_5Cu_{3.5}O_{10}$. They were synthetized by repeated heat treatment of well homogenized mixtures of reagent grade (p.a.) starting materials Y_2O_3, CuO, $BaCO_3$,′ La_2O_3 and $SrCO_3$, at 900–950°C, in air. Their X-ray diffraction patterns were used to estimate the degree of solid solubility in the crystalline phases resulting in the reactions between $YBa_2Cu_3O_{7-\delta}$ and La_2O_3 and $SrCO_3$, respectively.

The reaction products were characterized by their X-ray powder diffraction patterns obtained in Guinier–Hägg-type focusing cameras by using CuK_{α_1} radiation (λ = 1.54059 Å) and by SEM-EDS studies in a JEOL-820 scanning electron microscope equipped with a LINK AN-10000 EDS system using the ZAF program for evaluating the data.

Table 1 Crystalline compounds detected in the reaction products between YBCO and La_2O_3 (A) and YBCO and $SrCO_3$ (B)

r = MeO_n/YBCO	Crystalline phases detected*	
(A) *Me* = La		
0.1	M – Y_{123},	m – Y_{211}
0.2	M – Y_{123},	m – Y_{211}, m – LBC, m – La_{212}
0.4	M – Y_{123},	m – Y_{211}, m – LBC, m – La_{212}
1.0	M – Y_{123},	m – Y_{211}, m – LBC, M – La_{212}
2.0	t – Y_{123},	M – Y_{211}, M – LBC, M – La_{212}
3.0	–	m – La_{211}, M – LBC, m – La_{212}
(B) *Me* = Sr		
0.1	M – Y_{123},	m – 153.5
0.2	M – Y_{123},	m – 153.5
0.4	M – Y_{123},	M – 153.5, m – SBC, t – $(Ba_{1-z}Sr_z)CuO_3$
1.0	M – Y_{123},	M – 153.5, t – SBC,
2.0	m – Y_{123},	M – 153.5, m – SBC,
3.0		m – 153.5, M – SBC, t – $Y_2(Ba_{1-z}Sr_z)CuO_5$

* M=major, m=minor t=trace amounts,
LBC = $(La_{1-x}Ba_x)_2CuO_{4-\delta}$, with $x \approx 0.075 - 0.10$,
La_{212} = $La_{2-x}Ba_{1+x}Cu_2O_{6-\delta}$, with $x \approx 0.2 - 0.25$,
La_{211} = $La_{4-2x}Ba_{2-2x}Cu_{2-x}O_{10-2x}$, with $x \approx 0.0$,
SBC = $(Sr_{1-z}Ba_z)_2CuO_3$, with $z \approx 0.10$, and
153.5 = a phase with approximate chemical formula of $Y(Ba/Sr)_5Cu_{3.5}O_{10\pm\delta}$

Results and discussion

Crystalline compounds found in the reaction products according to the X-ray and SEM-EDS analyses are listed in Table 1. The cell parameters of the remaining Y_{123} were determined and listed in Table 2. In order to illustrate the effect of a possible substitution, the cell parameters of pure $YBa_2Cu_3O_{7-\delta}$, La-substituted $Y(Ba_{0.9}La_{0.1})_2Cu_3O_{7-\delta}$ and Sr-substituted $Y(Ba_{0.9}Sr_{0.1})_2Cu_3O_{7-\delta}$ (corresponding to a molar ratio $r=0.2$), respectively, are also listed.

Table 2 Cell parameters of the orthorhombic Y_{123}-phase in the reaction products between YBCO and $LaO_{1.5}$ (A), YBCO and $SrCO_3$ (B) and of some reference compounds (C)

r = MeO_n/YBCO	a/Å	b/Å	c/Å	V/Å^3
(A) *Me* = La				
0.1	3.8217 (7)	3.8862 (8)	11.672 (2)	173.36
0.2	3.8216 (7)	3.8869 (9)	11.676 (3)	173.44
0.4	3.8268 (5)	3.8873 (4)	11.674 (2)	173.66
1.0	3.8210 (2)	3.8910 (4)	11.640 (2)	173.19*
(B) *Me* = Sr				
0.2	3.8207 (8)	3.8865 (7)	11.675 (2)	173.36
0.4	3.8244 (7)	3.8840 (1)	11.678 (2)	173.50
1.0	3.8250 (1)	3.8880 (1)	11.680 (7)	173.75
(C)				
$YBa_2Cu_3O_{7-\delta}$	3.8240 (5)	3.8870 (5)	11.673 (2)	173.52
$Y(Ba_{0.9}La_{0.1})_2Cu_3O_{7-\delta}$	3.8285 (4)	3.8750 (7)	11.629 (2)	172.52
$Y(Ba_{0.9}Sr_{0.1})_2Cu_3O_{7-\delta}$	3.8125 (5)	3.8790 (4)	11.653 (3)	172.33

* Cell parameters determined from a limited number of diffraction lines

A. $YBa_2Cu_3O_{7-\delta}+La_2O_3$

Lanthanum oxide was found to react with $YBa_2Cu_3O_{7-\delta}$. Reactions between La_2O_3 and YBCO yielded $(La_{1-x}Ba_x)_2CuO_{4-\delta}$, $La_{2-x}Ba_{1+x}Cu_2O_{6-\delta}$ and La-substituted Y_2BaCuO_5 for $r=LaO_{1.5}/YBCO\leq 2.0$. The amount of the remaining, orthorhombic Y_{123}-phase decreased with increasing amounts of La_2O_3 and its cell parameters were found to remain practically unchanged (Table 2).

According to the data no substantial substitution of La for Ba in $YBa_2Cu_3O_{7-\delta}$ occurred, except for a possible slight doping of the remaining Y_{123}-phase at $r=1.0$. For $r=3.0$, the reaction yielded $(La_{1-x}Ba_x)_2CuO_{4-\delta}$, $La_{2-x}Ba_{1+x}Cu_2O_{6-\delta}$ and La_2BaCuO_5 with Y substituting partially for La.

The value of x in these compounds has been estimated by comparing the shifts in their X-ray diffraction lines with those of additionally prepared solid solutions, being about 0.075–0.10 in LBC and about 0.2–0.25 in La_{212}. These values of x were in agreement with those found by SEM-EDS measurements or the polycrystalline reaction products.

A slight shift of the strongest diffraction lines of the resulting Y_{211}-phase towards larger *d*-values suggests that the lanthanum ions substitute for the smaller yttrium and not for the larger barium ions. SEM-EDS analysis of the Y_{211}-phase in the reaction product for $r=1.0$ and 2.0 indicated an average formula of $(Y_{0.9}La_{0.1})_2BaCuO_5$. X-ray diffraction analysis of an additionally prepared solid solution series of samples of composition $(Y_{1-x}La_x)_2BaCuO_5$ confirmed the isomorphic substitution of Y by La up to $x\approx0.10$–0.15. The cell parameters of the resulting orthorhombic Y_{211} are listed in Table 3. For larger values of x polyphasic samples were obtained. On the other hand, attempts to prepare $Y_2(Ba_{1-x}La_x)CuO_5$, with $x=0.05$–0.2 have failed, the product obtained was found to be polyphasic, containing $La_4BaCu_5O_{13+\delta}$, unreacted Y_2O_3 and an orthorhombic La-substituted Y_{211}-phase.

Table 3 Cell parameters of the orthorhombic, La-substituted Y_{211} obtained from starting mixtures of composition $(Y_{1-x}La_x)_2BaCuO_5$

x in $(Y_{1-x}La_x)_2BaCuO_5$	a	b	c/Å	V/Å^3
$x=0.00$ (S)	7.1322(7)	12.179(1)	5.6597(7)	491.62
$x=0.05$ (S)	7.1395(8)	12.196(1)	5.6652(7)	493.28
$x=0.10$ (S)	7.1405(9)	12.200(2)	5.6664(9)	493.65
$x=0.15$ (P)	7.1480(2)	12.210(3)	5.6690(2)	494.82

S =single phase-, and P =polyphasic product.

B. $YBa_2Cu_3O_{7-\delta}+SrCO_3$

$SrCO_3$ was also found to react with $YBa_2Cu_3O_{7-\delta}$: $(Sr_{1-z}Ba_z)_2CuO_3$, Sr-substituted $Y_2(Ba_{1-z}Sr_z)CuO_5$, with $z\approx0.1$–0.15, trace amounts of Sr-substituted $(Ba_{1-z}Sr_z)CuO_2$, with $z\approx0.1$–0.2 (Table 1A) and – at least one – new phase resulted.

SEM-EDS analysis of Ba-substituted $(Sr_{1-z}Ba_z)_2CuO_3$-phase in the reaction product gave an average value of $z\approx0.1$. X-ray diffraction measurements of additionally prepared compounds wiht $z=0.0$ and 0.1 confirmed this observation: the cell parameters of orthorhombic $(Sr_{0.9}Ba_{0.1})_2CuO_3$ ($a=12.705(1)$, $b=3.9143(4)$, $c=3.5014(4)$Å and cell volume $V=174.14$Å^3), have been found to be slightly larger than those of the unsubstituted Sr_2CuO_3 ($a=12.7045(1)$, $b=3.9129(6)$, $c=3.4998(4)$Å and $V=173.98$Å^3).

Table 4 X-ray diffraction data for a compound with nominal composition $Y(Ba_{0.5}Sr_{0.5})_5Cu_{3.5}O_{10\pm\delta}$

Lattice spacings d/Å	Relative intensities
3.702	5
3.626	3
3.180	15
3.156	5
2.857	65
2.822	65
2.704	100
2.482	3
2.315	7
2.119	25
2.110	4
2.060	25
1.933	15
1.891	20
1.755	2
1.668	20
1.653	4
1.645	2
1.625	3
1.595	23
1.577	25

For values of $r=0.1-2.0$ the cell parameters of the Y_{123}-phase in the reaction products were found to be practically unchanged, indicating that the otherwise common Sr–Ba substitution did not take place this way.

A thorough SEM-EDS analysis of the polycrystalline reaction product between YBCO and $SrCO_3$ for $r=1.0$ revealed the presence of a crystalline phase with an approximate cationic ratio of $Y_{1.0(\pm0.1)}Ba_{2.5(\pm0.12)}Sr_{2.5(\pm0.20)}Cu_{3.5(\pm0.15)}$, which can be an oxide or, probably, an oxide-carbonate.

X-ray diffraction patterns of additionally prepared samples with the composition $YBa_{2.5}Sr_{2.5}Cu_{3.5}$-oxide were found to be similar with that of the unknown phase. A table of lattice spacings and relative intensities is given in Table 4 for identificational documentation. The exact composition and structure of this phase is the topic of ongoing work. This new phase is not identical with other previously reported Ba-rich phases: $YBa_3Cu_2O_{6+\delta}$, (Y_{132}), $YBa_4Cu_3O_{8.5+\delta}$ (Y_{143}) or $Y_{1.0-1.33}$ $Ba_8Cu_4(CO_3)_2O_{14.3}$ (Y_{184}).

Conclusion

Reactions between $YBa_2Cu_3O_{7-\delta}$ and La_2O_3 and $SrCO_3$, at 925°C in air, at ambient pressure, result in decomposition of the $YBa_2Cu_3O_{7-\delta}$. At values of $r = MeO_n/YBCO < 2.0$ the remaining $YBa_2Cu_3O_{7-\delta}$ was not found to be substituted neither by La nor by Sr.

Substantial La/Ba and Y/La and Ba/Sr intersolubility occurs in the crystalline reaction products. Reactions between $YBa_2Cu_3O_{7-\delta}$ and La_2O_3 yield $(La_{1-x}Ba_x)_2CuO_{4-\delta}$, with $x \approx 0.075$–0.10, $La_{2-x}Ba_{1+x}Cu_2O_{6-\delta}$, with $x \approx 0.2$–0.25 and $(Y_{1-x}La_x)_2BaCuO_5$, with $x \approx 0.1$–0.15 for values of $r \leq 2.0$. For $r = 3.0$ Y-substituted La_2BaCuO_5-phase was also detected in the reaction product.

Reactions between $YBa_2Cu_3O_{7-\delta}$ and $SrCO_3$ result in solid solutions of type $(Sr_{1-z}Ba_z)_2CuO_3$, with $z \approx 0.1$, $Y_2(Ba_{1-z}Sr_z)CuO_5$ with $z = 0.1 - 0.15$ and a compound with approximate composition of $Y(Ba_{0.5}Sr_{0.5})_5Cu_{3.5}O_{10\pm\delta}$.

* * *

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Zusammenfassung — Mittels Debye-Scherrer-Aufnahmen und Scanning-Elektronenmikroskopie wurden bei 925°C die Feststoffreaktionen zwischen dem Hoch-T_c-Keramiksupraleiter $YBa_2Cu_3O_{7-\delta}$ und La_2O_3 und $SrCO_3$ in Gemischen mit einem verschiedenen Molverhältnis $r = MeO_n/YBa_2Cu_3O_{7-x}$ untersucht. Die Reaktion zwischen $YBa_2Cu_3O_{7-\delta}$ und La_2O_3 ergibt $(La_{1-x}Ba_x)_2CuO_{4-\delta}$ mit $x \approx 0.075–0.10$, $La_{2-x}Ba_{1+x}Cu_2O_{6-\delta}$ mit $x=0.2–0.25$ und La-versetzte $(Y_{1-x}La_x)_2BaCuO_5$ mit $x=0.10–0.15$. Für $r=3.0$ entsteht außerdem noch Y-versetztes La_2BaCuO_5. Die Reaktion zwischen $YBa_2Cu_3O_{7-\delta}$ und $SrCO_3$ liefert $(Sr_{1-z}Ba_z)_2CuO_3$ mit $z \approx 0.1$, $Y_2(Ba_{1-z}Sr_z)CuO_5$ mit $z=0.1$-0.15 sowie eine nicht supraleitende Verbindung mit einer etwaigen Zusammensetzung von $Y(Ba_{0.5}Sr_{0.5})_5Cu_{3.5}O_{10\pm\delta}$. Bei Werten von $r \leq 2.0$ wurde unter den Reaktionsprodukten auch nicht umgesetztes $YBa_2Cu_3O_{7-\delta}$ gefunden.