Scientific Paper

SOL-GEL SYNTHESIS AND CHARACTERIZATION OF $YBa_2(Cu_{1-x}Cr_x)_4O_8$ SUPERCONDUCTOR

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Received 18-04-2004

Abstract

In this study the chromium substitution effects in the $YBa_2Cu_4O_8$ (Y-124) superconductor has been investigated. Superconducting oxide ceramics of composition $YBa_2(Cu_{1-x}Cr_x)_4O_8$ (x = 0.01; 0.03; 0.05; 0.10; 0.20) have been prepared by a simple aqueous sol–gel method using aqueous mixtures of the corresponding metal acetates and nitrates. Homogeneous precursor gels were obtained by complexing metal ions with tartaric acid. The obtained gel powders, intermediates and end products were characterized by X-ray powder diffraction analysis (XRD), infrared spectroscopy (IR), scanning electron microscopy (SEM) and resistivity measurements. The bulk samples were obtained after firing the precursor gels at 800 °C in flowing oxygen atmosphere. Doping the $YBa_2Cu_4O_8$ phase with chromium shows a strong effect on the superconducting properties decreasing the critical temperature from 78 K (for non-substituted sample) to 60 K (for the $YBa_2(Cu_{0.90}Cr_{0.10})_4O_8$). Synthesis product containing 20 % chromium instead of copper showed nonsuperconducting behaviour.

Key words: chromium, sol-gel, superconductor

Introduction

The search for high-temperature superconductivity and novel superconducting mechanisms is one of the most challenging tasks of material scientists.[1-4] The studies on various substitutions in oxide superconducting systems have been proven to be of great importance since the changes in critical transition temperature usually are observed. The $YBa_2Cu_4O_8$ (Y-124) superconducting compound is a member n = 2 of the homologous series of compounds of the general formula $Y_2Ba_4Cu_{6+n}O_{14+n}$ (n = 0, 1, 2).[5] The Y-124 phase is thermodynamically stable, isostructural, stoichiometric and thus is an ideal system to study different substitution effects.[6,7] For example, the effects of non-isovalent substitutions for Y or isovalent substitution for Ba in the Y-124 superconductor have attracted a great deal of attention in the past.[8-11] Doping of different ions at the copper sites in Y-124 superconductor serves as a useful diagnostic probe to investigate the role of different copper sites for the occurrence of superconductivity in these superconductors.[12-15]

In the context of doped materials, the incorporation of homogeneously distributed nanosized secondary phases in a host matrix which can be realized by the molecular level fabrication of new materials, is of significant interest.[16] Over the last few decades, sol-gel techniques have been used to prepare a variety mixed-metal oxides.[17-19] In these sol-gel processes, good quality of the oxide products was expected primarily due to the purity of the precursor materials used and chemical homogeneity obtained from the synthesis route.[20,21] Recently we described the use of an aqueous sol-gel method for the synthesis of superconducting Y-124 compound.[22,23] To date, no systematic studies have been reported on chromium substitution effects in the $YBa_2Cu_4O_8$ superconductor, to our knowledge. Such a situation has initiated the present work, motivating us to perform investigations on the effects of Cr substitution in the $YBa_2(Cu_{1-x}Cr_x)_4O_8$ superconductor.

Experimental

The $YBa_2(Cu_{1-x}Cr_x)_4O_8$ samples with x = 0.01, 0.03, 0.05, 0.10 and 0.20 were prepared by an acetate-tartrate sol-gel method. As starting compounds, stoichiometric amounts of Y_2O_3 , $Ba(CH_3COO)_2$, $Cu(CH_3COO)_2·H_2O$ and $Cr(NO_3)_3·9H_2O$, all of them analytical grade, were used. In the sol-gel process Y_2O_3 first was dissolved in 0.2 M acetic acid at 55-60 °C. Next, $Ba(CH_3COO)_2$ and $Cu(CH_3COO)_2·H_2O$ or appropriate mixtures of $Cr(NO_3)_3·9H_2O$ and $Cu(CH_3COO)_2·H_2O$, all of them dissolved in a small amount of distilled water, were added with intermediate stirring during several hours at the same temperature. The obtained solutions were concentrated during about 8 hours at 60-65 °C in an open beaker. Under continuous stirring the transparent blue gels have formed. After further drying in an oven at 80 °C fine grained blue powders were obtained. The precursor gel powders were calcined for 10 hours at 700 °C in flowing oxygen, reground in an agate mortar, and again heated for 20 hours at 800 °C again reground, pelletized and annealed for 20 hours at 800 °C in a flowing oxygen atmosphere at ambient pressure.

The synthesized samples were characterized by X-ray powder analysis performed with a Siemens D-500 diffractometer, using $CuK\alpha_1$ radiation. For IR studies, a Perkin-Elmer FT-IR Spectrum BX II apparatus was used. The samples were mixed (1.5%) with dried KBr and pressed into pellets. Scanning electron microscope (SEM) JEOL 820 was used to study the morphology of the samples. Superconducting properties were obtained by resistivity measurements using a standard four-point technique.

Results and Discussion

Characterization of the gel precursors

The special attention was paid to the powder X-ray diffraction studies of the gel precursors. The XRD patterns of the Y-Ba-Cu(Cr)-O acetate-tartrate gels are shown in Figure 1. As seen, the XRD patterns of the obtained Y-Ba-Cu(Cr)-O gels show broad peaks due to the amorphous character of the powders. It has been observed that, if the conditions of the sol-gel process are not optimized, partial crystallisation of initial metal salts may occur.[24] However, no peaks due to insignificant crystallisation of metal acetates, tartrates, or nitrates, or crystallisation of any undesired or contaminating phase could be identified. Such observation shows the individuality and high level of homogeneity of the synthesized Y-Ba-Cu(Cr)-O precursor gels.

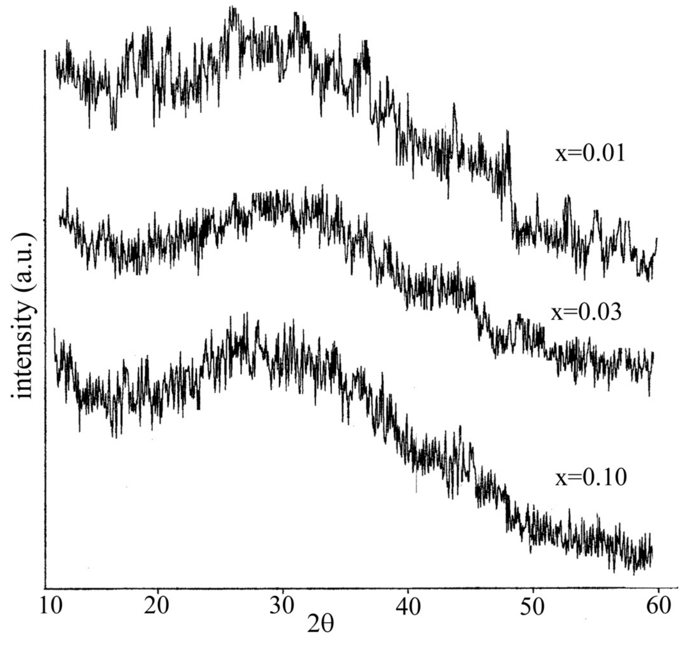


Figure 1. X-ray diffraction patterns of the Y-Ba-Cu(Cr)-O precursor gels for the preparation of $YBa_2(Cu_{1-x}Cr_x)_4O_8$ with different amount of Cr.

Infrared (IR) spectra of substituted Y-Ba-Cu(Cr)-O precursor gels were measured as well. All spectra for the samples with low amount of Cr qualitatively were identical regardless of chromium substitutional level. The IR spectra of two representative samples (gels with 1% and 20% of Cr substitutional level) are presented in Figure 2. Since -CH_3, >CH_2, -CO-OH and >CH-OH stretchings could be easily identified in all IR spectra,[20] we suppose that both acetate and tartrate ligands are in the coordination sphere of metals. The specific peaks observed in the 850-500 cm^{-1} region of the IR spectra (823, 788, 762, 680, 655 and 618 cm^{-1}) may be attributed to characteristic M-O vibrations.[25] As already mentioned, Y-Ba-Cu(Cr)-O gels with low substitutional level of Cr (1%, 3%, 5% and 10%) showed almost identical characteristics vibrations. However, some specific changes in the IR spectrum of Y-Ba-Cu(Cr)-O gel with 20% of Cr (see Figure 2) are evident. The small band at 1777 cm^{-1} can be assigned to the nitrate groups. The splitting of the absorption band in the 1430-1350 cm^{-1} region is also present in the spectrum. Absorption at 1416 cm^{-1} is originated from stretch vibrations in CH_2 and CH_3, and absorption at 1360 cm^{-1} can be assigned to NO_3^-. Therefore, we can state that chromium nitrate is present in the Y-Ba-Cu(Cr)-O gel synthesized with higher amount of Cr (20%). Since chromium nitrate peaks were not identified in the XRD pattern, it is possible to conclude that the crystallization of initial material during gelation is negligible.

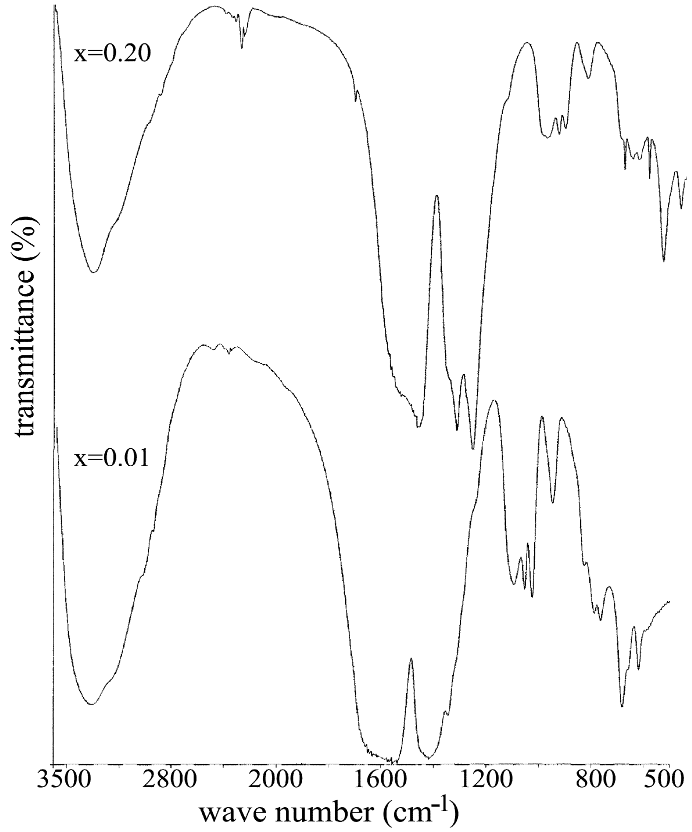


Figure 2. IR spectra of the Y-Ba-Cu(Cr)-O acetate-tartrate gels with different substitutional level of Cr instead of Cu.

The morphological features of the Y-Ba-Cu(Cr)-O gels are almost identical regardless of Cr substitutional level. The typical scanning electron photograph is shown in Figure 3.

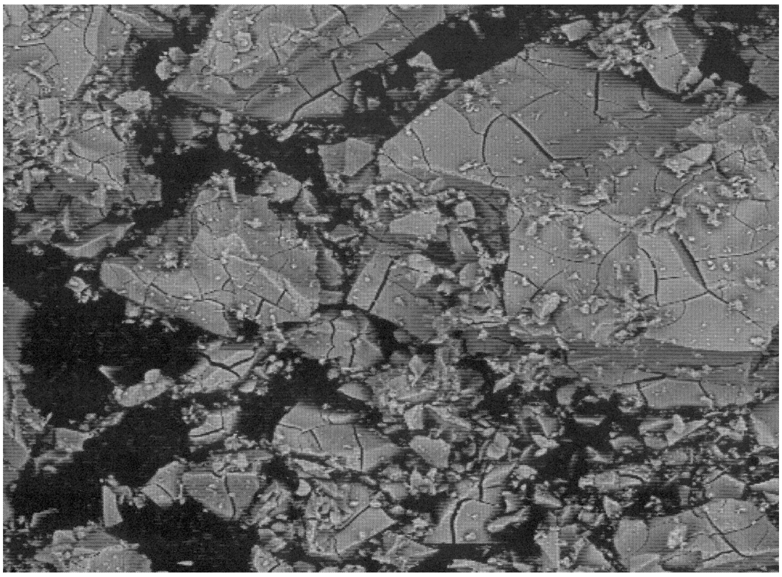


Figure 3. SEM micrograph of the Y-Ba-Cu(Cr)-O acetate-tartrate gel (5 % Cr). Magnification: 250 X.

As seen, a monolithic gel structure is formed, keeping a uniform network with no internal porosity.

Characterization of the intermediates

Characterization of the intermediates is interesting from the theoretical point to understand the possible mechanisms of the formation of desired superconducting phase. For example, the phase evolution and formation mechanisms of pure Y-124 compound synthesized by acetate-tartrate sol-gel technique have been well documented elsewhere.[26,27] Therefore the Y-Ba-Cu(Cr)-O precursor gels were calcined for 10 h at 700 °C in flowing oxygen atmosphere. The same physical methods were used for the characterization of the obtained intermediates. The X-ray diffraction patterns of two representative samples are shown in Figure 4. As seen, the XRD patterns of intermediates are rather complicated indicating the formation of multiphasic products. To compare these results with literature data,[8-11] it is clear that no even traces of superconducting Y-124 phase have formed during the initial calcination of Y-Ba-

Cu(Cr)-O precursor gel samples. Annealing at 700 °C produced a wide variety of mixed-metal oxides ant carbonates having different chemical composition.

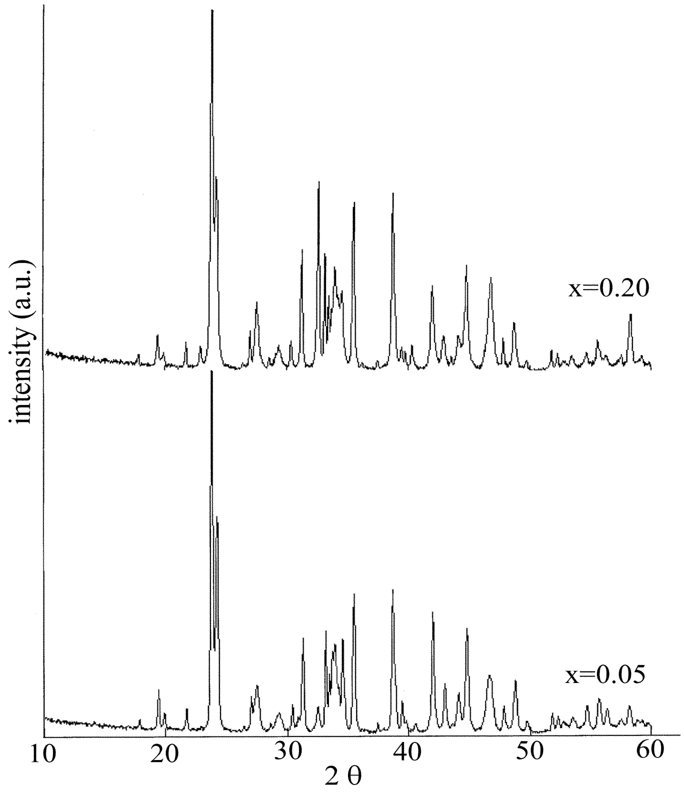


Figure 4. XRD patterns of two Y-Ba-Cu(Cr)-O precursor gel samples calcined at 700 °C.

For instance, the X-ray diffraction peaks around 2θ = 27-29° represent the most intensive diffraction lines of $BaCuO_2$ and $Ba_2Cu_3O_{5+x}$ (PDF 38-1402 and PDF 40-312, respectively). According to JCPDS© 1991 and 1996 reference data, in the region of 2θ = 29-40° most of the peaks could be attributed to the tetragonal $YBa_2Cu_3O_{7-\delta}$ (Y-123) and $Y_2Ba_4Cu_7O_{15}$ (Y-247) phases. The main diffractions of $Ba_4Y_2O_7$ (PDF 28-0163) and CuO (PDF 5-661) phases are located in this region as well. The most intensive peaks presented in the XRD patterns at around 2θ = 24-25° indicate the presence of big amount of unreacted $BaCO_3$ (PDF 5-378) in the intermediate product. The identification of few peaks, however, were found to be problematic. From XRD analysis data it is possible to presume that the formation of Y-124 phase occurs via initial formation of first two members (Y-123 and Y-247) of homologous series of $Y_2Ba_4Cu_{6+n}O_{14+n}$ and further reaction with secondary phases at higher temperatures.

The IR spectra for the intermediates were also recorded. Again, IR spectra for different specimens were almost identical. The IR spectra for two representative samples are shown in Figure 5.

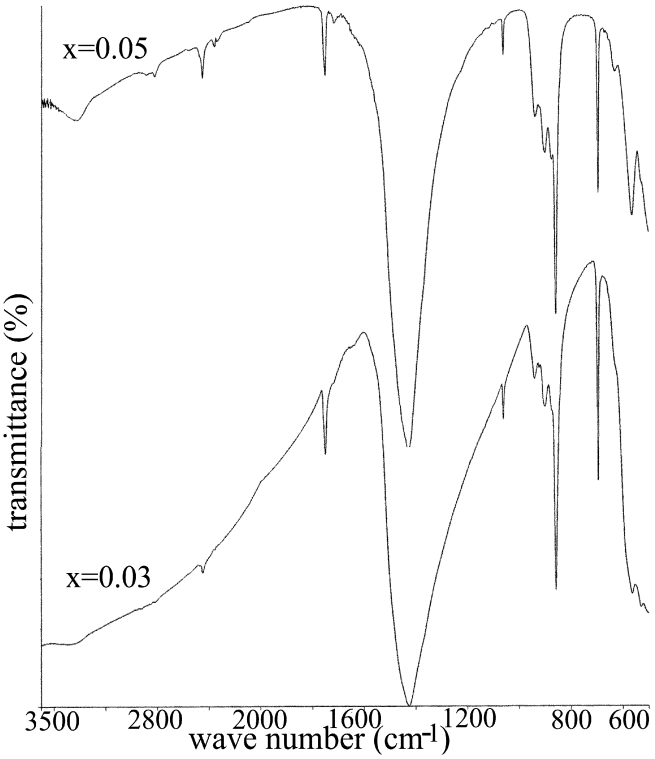


Figure 5. IR spectra of two Y-Ba-Cu(Cr)-O precursor gel samples calcined at 700 °C.

In the IR spectra of Y-Ba-Cu(Cr)-O samples annealed at 700 °C the bands at 1750, 1440 and 860 cm^{-1}, assignable to carbonates[28,29] predominate. The bands in the region of 800-560 cm^{-1} are the characteristics metal-oxygen vibrations.[30]

The textural properties of the intermediate samples were also investigated by SEM. The SEM micrographs showed that intermediates are formed from spherical particles, plate-like grains and needle-like micrograins. The formation of differently shaped crystallites with an average grain size ranging from 1 μm to 10 μm was evident from these investigations. Figure 6 shows SEM micrograph (back scattered electron (BSE) image) of Y-Ba-Cu(Cr)-O powders calcined at 700 °C.

Since the brightness of the specimen is highly inhomogeneous over the entire measuring area, most of the material is not finely divided, i.e. the distribution of its chemical elements is not uniform. Such observations support previous results obtained by XRD and IR analyses. The additional homogenization of the intermediates and further heating at higher temperatures are necessary to get $YBa_2(Cu_{1-x}Cr_x)_4O_8$.

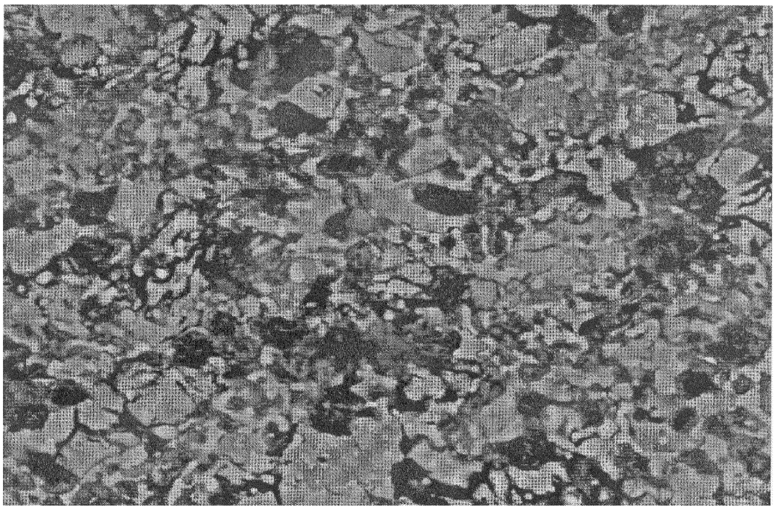


Figure 6. Scanning electron micrograph (in back-scattered electron mode) of Y-Ba-Cu(Cr)-O precursor gel sample (x = 0.03) calcined at 700 °C. Magnification: 1000 X.

Characterization of the end products

Figure 7 shows the XRD patterns of the $YBa_2(Cu_{1-x}Cr_x)_4O_8$ samples with 1% and 3% of Cr substitutional level.

As seen, the XRD patterns for chromium-substituted $YBa_2(Cu_{0.99}Cr_{0.01})_4O_8$ and $YBa_2(Cu_{0.97}Cr_{0.03})_4O_8$ samples are almost identical. According to X-ray diffraction analysis almost monophasic Y-124 materials were obtained in both of the cases. Only minor amount of $Ba_4Y_2O_7$ has formed ($2\theta = 29°$) as impurity phase. The patterns of the chromium-substituted Y-124 samples, however, showed considerable changes upon increasing substitution. The X-ray diffraction pattern of the specimen with 5% of Cr is shown in Figure 8. The characteristic features of the orthorhombic Y-124 diffraction pattern already vanish: the intensity of the (113) and (108) diffractions at $2\theta \cong 34.4°$ and 35.8° decreases strongly and the splitting of the (200) (020) (0014) at $2\theta \cong 47°$ and the (017) (111) diffractions at $2\theta \cong 33°$ vanishes. Moreover, an additional not intensive diffraction line at $2\theta = 33.6°$ due to the presence of Cr_2O_3 (PDF 38-1479), and two more intensive peaks around $2\theta = 21.2°$ and 25.9°, which are the most intensive (90% and 100%) lines for CrO_3 (PDF 32-285) were observed in the XRD pattern.

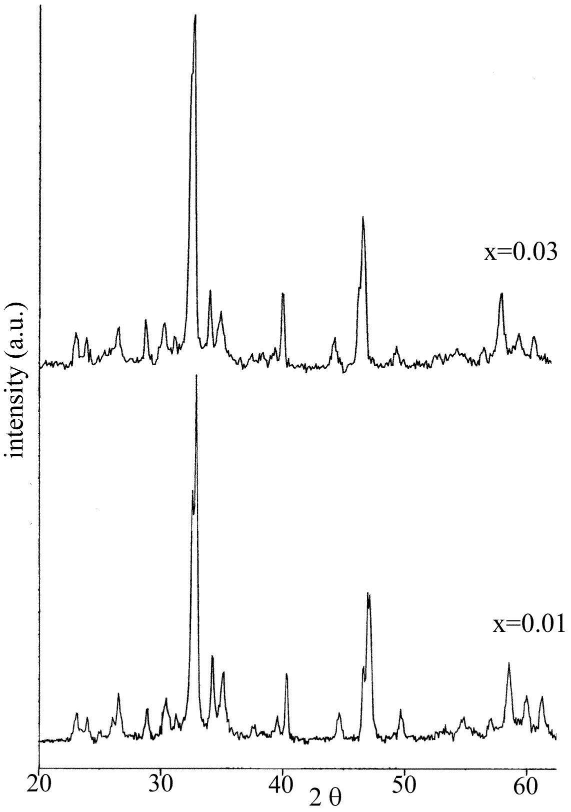


Figure 7. Powder X-ray diffraction patterns of the $YBa_2(Cu_{1-x}Cr_x)_4O_8$ samples with x = 0.01 and x = 0.03.

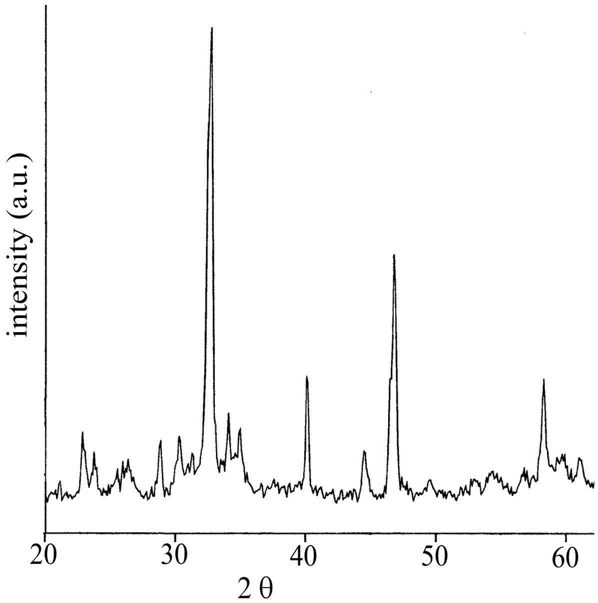


Figure 8. Powder X-ray diffraction pattern of the $YBa_2(Cu_{1-x}Cr_x)_4O_8$ samples with x = 0.05.

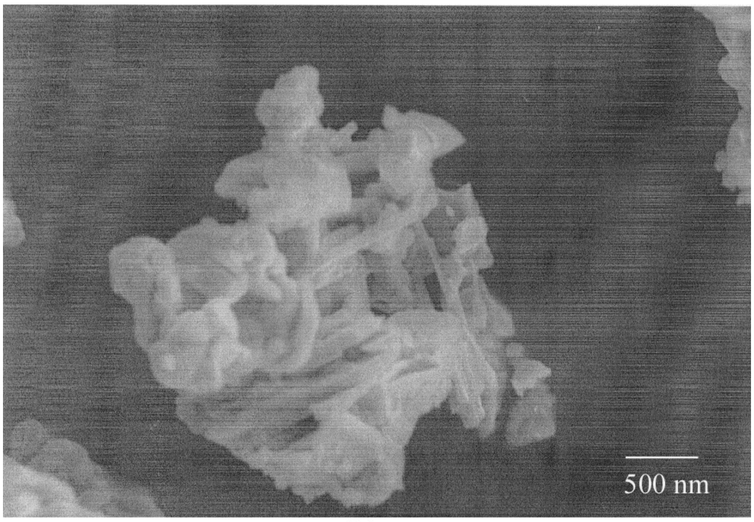
The XRD pattern of $YBa_2(Cu_{0.90}Cr_{0.10})_4O_8$ sample is comparable with previous one presented in Figure 8. The sample contains Y-124 phase, and Cr_2O_3 and CrO_3 as well. However, peak intensities of the impurity phases increased considerably. On further increasing the substitution level a main peak in the XRD patterns of these samples can be attributed already to the impurity phase. Moreover, in the case of x = 20 the peaks due to the Y-124 phase have disappeared. The XRD pattern contains predominating tetragonal Y-123 phase and no traces of $YBa_2(Cu_{0.80}Cr_{0.20})_4O_8$ phase has been detected.

The carbonate peaks which have been indicated for the intermediates (1750, 1440 and 860 cm^{-1}), are not visible anymore in the IR spectra of end products. This indicates that the decarbonization of the carbonate and oxicarbonate phases have fully proceeded at higher temperature.

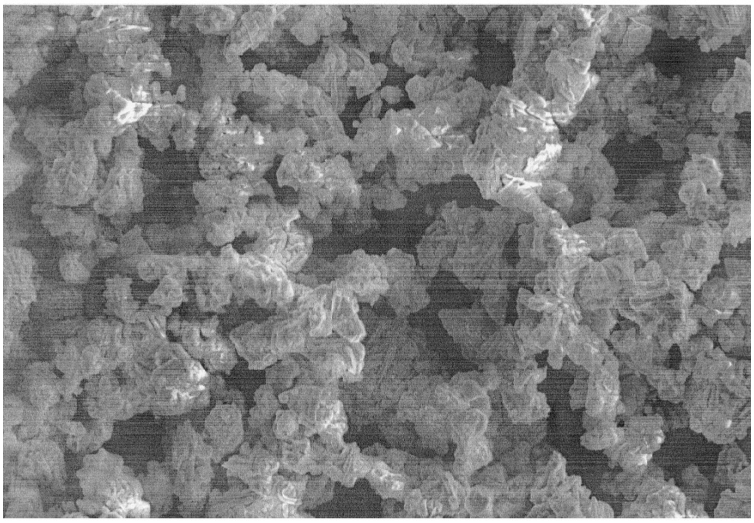
A progressive change in morphology is evident with increased calcination temperature. Figure 9 shows the surface features of the chromium-substituted $YBa_2(Cu_{0.99}Cr_{0.01})_4O_8$ powders calcined at 800 °C.

From Figure 9b is seen that chromium-substituted Y-124 solids are composed of volumetric grains with no regular shape. For example, the plate-like grains coexist with spherically shaped particles which size is ranging from 1 to 5 μm (see Figure 9a). It is interesting to note, however, that the particle size does not increase with firing temperature.

Few studies on the investigation of the substitution of chromium for copper in other cuprate superconductors have been reported in the past. Abdshukor et al.[31] for example, found that the onset superconducting transition temperatures of $ErBa_2(Cu_{1-x}Cr_x)_2Cu_3O_{7-\delta}$ ($0 \leq x \leq 0.1$) (Er-123) samples were nearly constant within the substitution range. However, the transition width broadened as the Cr content was increased. On the other hand, Ilonca et al.[32] have demonstrated that the transport coefficients in the substituted $(Bi_{1.6}Pb_{0.4})(Sr_{1.8}Ba_{0.2})Ca_2(Cu_{1-x}Cr_x)_3O_y$ ($0 \leq x \leq 0.07$) (Bi-2223) depend strongly on the chromium content in the samples. Snowling and Grovenor[33] have also reported that T_C of $TlSr_2Ca_{1-x}Cr_xCu_2O_{7+\delta}$ superconducting sample does not vary with adding of 0.5 mol equivalent of CrO_3 during sintering of Tl-1212 phase. However the transition was found to be sharper to compare with non-substituted sample. The electrical properties of $YBa_2(Cu_{1-x}Cr_x)_4O_8$ samples are summarized in Table 1.



(a)



(b)

Figure 9. SEM micrograph of the $YBa_2(Cu_{0.99}Cr_{0.01})_4O_8$ superconductor at two magnifications: (a) 3500 X and (b) 1000 X.

Table 1. Superconducting properties of $YBa_2(Cu_{1-x}Cr_x)_4O_8$ samples.

Amount of Cr (%)	T_C (onset)/K	T_C (zero)/K	ΔT_C/K
1	70	57	13
3	68	53	15
5	65	48	17
10	60	41	19
20	–	–	–

$YBa_2(Cu_{1-x}Cr_x)_4O_8$ samples with x = 0.01, 0.03, 0.05 and 0.10 are superconducting, and the last sample with x = 0.20 is not superconducting.

Previously[22,23] we have reported that non-substituted sol-gel derived Y-124 superconductor exhibited a sharp superconducting transition with T_C (onset) = 78 K and T_C (zero) = 67 K. Thus, with chromium substitutions in small concentrations up to 10%, the effect on superconductivity is however deleterious with T_C getting suppressed at a rate of about 2 K per %. The onset critical temperature of superconductivity decreased down to 60 K for $YBa_2(Cu_{0.90}Cr_{0.10})_4O_8$ (T_C (zero) = 41 K), and the transition range becoming broader with increasing substitution. The $YBa_2(Cu_{1-x}Cr_x)_4O_8$ samples with x = 0.20 exhibited only semiconducting behaviour indicating the loss of superconductivity at 20% Cr doping. In conclusion, the less dramatic chromium substitution effect on T_C compared with Mn-substituted,[15] Fe-substituted,[34] Co-substituted,[35] Ni-substituted,[35] and Zn-substituted[36] Y-124 samples was rather unexpected. The most interesting fact is that compound with 10% of Cr ($YBa_2(Cu_{0.90}Cr_{0.10})_4O_8$) still is superconducting. This can be explained perhaps in two ways: (i) co-existence of isovalent pair of Cr^{6+}/Cr^{3+} or (ii) the complete substitution by Cr at Cu sites does not proceed. To prove such hypotheses the further studies (for instance, neutron powder diffraction analysis and X-ray photoelectron spectroscopy) of $YBa_2(Cu_{1-x}Cr_x)_4O_8$ samples probing whether Cr doping causes a charge transfer to CuO_2 layers[37] and structural changes, such as the axial Cu-O bond length as well as the Cu-Cu distance,[38] could be done.

Conclusions

Cr-substituted $YBa_2(Cu_{1-x}Cr_x)_4O_8$ samples were synthesized by a simple aqueous sol-gel method. Y-Ba-Cu(Cr)-O acetate-tartrate gels, the products after intermediate calcination of precursor gels at 700 °C and the end products obtained at 800 °C were characterized by powder X-ray diffraction analysis, infrared spectroscopy and scanning electron microscopy. The gels synthesized were found to be amorphous, highly homogeneous and individual compounds. We have demonstrated that Y-124 phase did not form after calcinations of gels at 700 °C. Annealing at this temperature produced a wide variety of different oxide and carbonate phases. XRD measurements indicated that in $YBa_2(Cu_{1-x}Cr_x)_4O_8$ samples (up to x = 0.10) synthesized at 800 °C the crystal structure remains orthorhombic. With further increasing the chromium substitutional level peaks due to the Y-124 phase disappeared. We have also demonstrated, that the critical temperature of superconductivity decreased from 78 K for non-substituted sample to 60 K for the sample with 10% of chromium substitutional level. With further increasing the chromium substitutional level, for instance the sample with x = 0.20 has lost its superconductivity. The observations showed that T_C of the $YBa_2(Cu_{1-x}Cr_x)_4O_8$ decreases almost linearly with increasing x. The rate of T_C suppression was determined to be approximately 2 K per %.

Acknowledgements

The financial support from the Lithuanian State Science and Education Foundation under project MODELITA (No. C-03048) is gratefully acknowledged.

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Povzetek

V superprevodniku $YBa_2Cu_4O_8$ (Y-124) smo z zamenjavo dela bakra s kromom pripravili $YBa_2(Cu_{1-x}Cr_x)_4O_8$ (x = 0.01; 0.03; 0.05; 0.10; 0.20) s sol-gel metodo iz vodnih raztopin ustreznih acetatov in nitratov z dodatkom vinske kisline. Produkte smo karakterizirali z rentgensko praškovno analizo (XRD), infrardečo spektroskopijo (IR), elektronsko mikroskopijo (SEM) in meritvami prevodnosti. Dopiranje $YBa_2Cu_4O_8$ s kromom zniža kritično temperaturo od 78 K (za nedopiran vzorec) na 60 K (za $YBa_2(Cu_{0.90}Cr_{0.10})_4O_8$). Produkt, ki ima 20% bakra zamenjanega s kromom, nima več superprevodnih lastnosti.