Effect of Irradiation on the Superconductor Properties of Bi$_2$Sr$_{2-x}$Li$_x$Ca$_2$Cu$_3$O$_y$

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Abstract

High temperature superconductor Bi$_2$Sr$_{2-x}$Li$_x$Ca$_2$Cu$_3$O$_y$ was prepared by a solid state reaction method, with firing temperature 800 °C for 24 hours and sintering temperature 850 °C for 140 hours. Superconductor properties studied before and after irradiation, critical temperature and oxygen content increases by increasing of ratio of Li (Li=0.1, 0.2, 0.3), but its decrease after irradiation.

X-ray diffraction analysis showed that the compounds have two phases: high-$T_c$ phase (2223) and low-$T_c$ phase(2212) in addition to impurity phase, also irradiation lead to decreases volume fraction of high-$T_c$ phase (2223) and appear new peaks.

Scanning electron microscope has been used to evidence the morphology of the superconductor phases. Grain size increases by increasing of Li concentration and these grains take square-shaped, but after irradiation grain size becomes smaller and square - shaped disappear.

الخلاصة

حضرت نماذج من المركب Bi$_2$Sr$_{2-x}$Li$_x$Ca$_2$Cu$_3$O$_y$ حرارة حرق 800 °C لمدة 24 ساعة ودرجة حرارة تبلứt 850 °C لمدة 140 ساعة، درست خواص أموصل ألفائق أنتوسيبول بطريقة تفاعل أحاله أصلبه ودرجة ألفائق قبل وبعد التشعيب، أظهرت تحليلات أشعة ألسن ربية إن المركب يمتلك طورين: ألفائق أنتوسيبول أفعال (2223) والطور ألفائق التوصيل الواطي (2212) بالإضافة إلى طور الشوائب، كذلك وجد أن التشعيب يؤدي إلى تقليل النسبة ألحجمي للطور ألفائق التوصيل العالي (2223) وظهور قم جديدة.

لقد استخدم المجهر الإلكتروني الماسح لدراسة تشكيلة الأطور فائقه ألفائق التوصيل وقد وجد ان الحجم الحبيبي يزداد بزيادة تركيز أليليوم وهذه الحبيبيات تكون مربعة الشكل ولكن بعد التشعيب يصبح الحجم الحبيبي أصغر ويخفي الشكل المربع للحيبيات.
Introduction

Superconductors are materials whose resistance is zero below a certain temperature, called the critical temperature ($T_c$) (Maqsood and Maqsood, 1996), this material including simple elements, metallic alloys and certain ceramic compounds, these compounds known as the cuprate high temperate superconductor (Hott et al., 2004).

Based on the number of CuO$_2$ planes in the characteristic multilayer blocks, the high critical temperature cuprate superconductors can be classified into: single layer materials [e.g., Bi$_2$Sr$_2$CuO$_{6+\delta}$ (Bi2201)], bilayer materials [e.g., Bi$_2$Sr$_2$Ca-Cu$_2$O$_{8+\delta}$ (Bi2212)] and trilayer materials [e.g., Bi$_2$Sr$_2$Ca$_2$Cu$_3$O$_{10+\delta}$ (Bi2223)] (Damascelli et al., 2002). This structural characteristic has a direct correlation with the superconducting properties: within each family of cuprates, the superconducting phase transition temperature ($T_c$) first increases with the layer number ($n$) (Bernhard et al., 2004). Because of the highest critical temperature, the (2223) phase of the compound has attracted greater interest and many workers have spent much effort to obtain the pure phase of the compound in the bulk, this phase has unstable structure (Ertan and Necdet, 2001). Intergrowth of multiple phases in crystal grain has posed difficulties in preparing single phase crystals (Shaoyan and Michal, 1998).

These compound are very important practical applications, therefore we suggested that study the effect of gamma radiation on the superconductor properties.

Experimental Procedure

The samples were prepared by standard solid state reaction from the starting materials Bi$_2$O$_3$, Sr(NO$_3$)$_2$, LiCo$_3$, CaCo$_3$ and CuO. These were mixed and the mixture was thoroughly ground in an agate mortar and then calcined at 800 °C for 24 hours in a furnace to remove CO$_2$ and NO$_2$ gases from the mixture.

The calcined powder was reground again, pressed to pellets with (1.2cm) in diameter under a hydrostatic pressure about (0.5Gpa).The pellets were sintered at 850 °C for 140 hours. The pellets were examined by Miessner effect to evaluate the superconducting state.

Oxygen content in the samples was determined by idometric titration method, these method include: weighting (45mg) of sample and placed inside a conical
flasks on the magnetic stirrer, adding (2.5ml) of KI solution and (1.25ml) of HCl solution are added together to the samples powder, the liquid in the flask turns dark brown, the solution of Na$_2$S$_2$O$_3$ is added to the liquid from the burette when the liquid becomes pale brown a few drops of starch were added. The liquid turns dark blue, further addition of the Na$_2$S$_2$O$_3$ solution with more slowly will turn the liquid to yellow. Stopped the titration and measured the volume of the titrated Na$_2$S$_2$O$_3$ solution, and oxygen content ($\delta$) could be found from equation (Swinnea et al., 1987).

\[
\delta = \left(\frac{M_A}{M_B} - \frac{3m_A}{CV}\right)/\left(\frac{2m_A}{CV} - \frac{M_0}{M_B}\right)
\]

(1)

where $M_A$ is the molar mass of the sample, $m_B$ molar masses of Na$_2$S$_2$O$_3$.5H$_2$O, $m_A$ weight of the sample, C concentration of the Na$_2$S$_2$O$_3$, V volume of Na$_2$S$_2$O$_3$ used in titration and $M_0$ atomic weight of oxygen.

The phase compositions of samples were analyzed by X-ray diffraction analysis using Cu-Kα radiation. The electrical properties were measure by four-point probe method, in the temperature intervals of (77-300) K and direct current from 0.5mA to 15mA was admitted throw the sample. The resistivity could be found from the relation (Sorab, 1968).

\[
\rho = \frac{VWt}{IL}
\]

(2)

where I is the current passing throw the sample, V the voltage drop across the electrodes, W width of the sample, t thickness of the sample and L the effective length between the electrodes. The microstructure of the sample was observed using a scanning electron microscopy (SEM); the observation was performed on the surface of the samples.

Samples have been irradiated by gamma ray at room temperature by using Co$^{60}$, the test samples were irradiated dose 23MRad, all these samples have been examined before and after irradiation.
Results and Discussion

The values of excess oxygen content are shown in Table (1). It could be noted that the value of excess oxygen content ($\delta$) of the specimens doping with lithium gradually increases because of the doping introduce additional oxygen in to the Li/Sr-O planes. After irradiation the amount of oxygen is decreases because irradiation act on the damage the bonds in the CuO planes and consist of defects, these defects decreases the number of holes in the lattice (Bodi et al., 2001).

Table (1). Oxygen content of Bi$_{2}$Sr$_{2-x}$Li$_{x}$Ca$_{2}$Cu$_{3}$O$_{Y}$ before and after irradiation.

<table>
<thead>
<tr>
<th>$x$</th>
<th>Oxygen content</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Before irradiation</td>
</tr>
<tr>
<td>0.1</td>
<td>0.213</td>
</tr>
<tr>
<td>0.2</td>
<td>0.259</td>
</tr>
<tr>
<td>0.3</td>
<td>0.324</td>
</tr>
</tbody>
</table>

On the other hand, the critical temperature of the prepared sample increasing by increasing of lithium concentration ($T_c=102,110,120$)K when ($x=0.1,0.2,0.3$) respectively, as shown in figure (1) before irradiation, apparently it is due to the fact that, for small heterovalent replacement of 2-valent copper cation by 1-valent lithium ion, the concentration of Cu$^{+3}$ holes in the CuO$_2$ layers increases (Meretliev et al., 200).The critical temperature of the samples decreased after irradiation, where taken the values ($T_c=93,102,109$)K when ($x=0.1,0.2,0.3$) respectively, see figure (2), due to change in oxygen stoichemetry, also this may be attributed to the radiation induce damage of weak links which result in poor grain conductivity in Bi-2223 compound (Borisenko et al., 2006).
Figure (3) shows x-ray diffraction of the prepared samples before irradiation, we could be seen from the spectra that two phases in all samples: high-\(T_c\) phase (2223), low-\(T_c\) phase and a small amount of impurity phases. The substitution of Sr by Li may well relax the modulation by influencing the charge balance oxygen content and structure of the relevant layers (Mazaki et al., 2005), from these figure can be noticed that the high rate of (2223) phase appears with increasing Li content and slightly change in the intensity. Figure (4) shows x-ray diffraction of irradiated samples, from this figure observe the volume fraction of high-\(T_c\) phase decreasing and the irradiation cause shifted of peaks, as well as some oxide impurities were found to be developed peaks corresponding to another oxide impurities which may be attributed to the absent of some oxygen atoms due to irradiation (Kivelson et al., 2006).

Figure (5) shows SEM photographs of samples before irradiation. It can be observed from figure (5a) plate-link grains which consist of 2212 phase and grain boundaries are very clean. From figure (5b) we can see that some small particles are seen on the edge of the plate-like, the size and amount of particle increase and the shape of these particles is square (figure (5c)) (Guo, et al., 2000).

Figure (6) shows SEM of irradiated samples, from this figure we can see these particles become smaller and less continuously and the square shaped particles disappeared (Ramesh, 2005).
Figure (1). Temperature dependence of resistivity of Bi$_2$Sr$_{2-x}$Li$_x$Ca$_2$Cu$_3$O$_y$ before irradiation

Figure (2). Temperature dependence of resistivity of Bi$_2$Sr$_{2-x}$Li$_x$Ca$_2$Cu$_3$O$_y$ after irradiation
Figure (3). X-ray diffraction of Bi$_2$Sr$_{2-x}$Li$_x$Ca$_2$Cu$_3$O$_y$ before irradiation.
Figure (4). X-ray diffraction of Bi$_2$Sr$_{2-x}$Li$_x$Ca$_2$Cu$_3$O$_{y}$ after irradiation.
**Figure (5).** SEM of Bi$_{2}$Sr$_{2-x}$Li$_{x}$Ca$_{2}$Cu$_{3}$O$_{Y}$ before irradiation.
Figure (6). SEM of Bi$_{2}$Sr$_{2-x}$Li$_{x}$Ca$_{2}$Cu$_{3}$O$_{Y}$ after irradiation.

Conclusions

The critical temperature increases by increasing of Li, also excess oxygen content, while its decreasing for irradiated samples, as well as irradiation leads to decreasing in volume fraction of high-$T_c$ phase with appearing of new peaks of impurity oxide phases. Scanning electron microscope obvious plate-like grains which consist of (2212) phase and the shape of particles is square but these particles become small and the square-shaped faded after irradiation.
References


