

# Effect of K Partial Substitution on the Mechanical Properties of Bi-2223 Superconductors

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Electrical and mechanical properties of the  $\text{Bi}_{2-x}\text{K}_x\text{Pb}_{0.3}\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_y$  superconductors for  $0 \leq x \leq 0.5$  was investigated using the dc electrical resistivity and static Vickers hardness tests. The results show improvements in the superconducting and deterioration of the mechanical properties with increasing the substitution, as confirmed by X-ray diffraction and scanning electron microscope investigations.

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PACS/topics: mechanical properties, Bi-based superconductors, K substitution

## 1. Introduction

The better of understanding the physics of superconductivity and appearance of new applications depend strongly on the development in materials research, which is still in progress. It has been found that superconducting properties of  $\text{BiSrCaCuO}$  (BSCCO) compounds can be enhanced by the substitution or addition of the elements with different ionic radii and different bonding characters to these compounds [1, 2]. Investigation of the mechanical properties of BSCCO compounds is important for developing their applications. A number of studies have been carried out to improve the mechanical properties of superconductors [3, 4]. In this work we are trying to investigate the effect of sintering temperature and partial substitution of K on Bi sites on the electrical and mechanical properties of the samples.

## 2. Materials and equipment

The  $\text{Bi}_{2-x}\text{K}_x\text{Pb}_{0.3}\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_y$  samples with ( $x = 0.0 - 0.5$ ) were synthesized by the solid state reaction method. Molar ratios of the high purity powders of  $\text{Bi}_2(\text{CO}_3)_3$ ,  $\text{K}_2\text{CO}_3$ ,  $\text{Pb}_3\text{O}_4$ ,  $\text{Sr}(\text{NO}_3)_2$ ,  $\text{CaCO}_3$  and  $\text{CuO}$  were well mixed. The mixture was calcined in air at  $800^\circ\text{C}$  for 24 h. The powder was pressed into pellets under pressure around 0.5 GPa. All samples were sintered in air for 140 h at different sintering temperatures of  $830^\circ\text{C}$ ,  $840^\circ\text{C}$ ,  $850^\circ\text{C}$  and  $860^\circ\text{C}$ .

The resistivity measurements were performed by the standard four-probe method on all samples. X-ray diffraction (XRD), measured using a Philips diffractometer with  $\text{Cu K}\alpha$  radiation, was used to investigate the structural characteristics of the samples. Scanning electron microscopy (SEM type TESCAN) was used to analyze the surface morphology of samples. Hardness measurements of the samples were performed on the polished surfaces of the examined samples with a digital microhardness tester (HVS-1000) at room temperature. The

applied load  $F$  was varied in the range of 0.245–2.940 N and was applied for 20 seconds. The load dependent (Vickers) microhardness values, Young modulus  $E$  and yield strength  $Y$  of the samples are calculated using the relations from Refs. 5, 6 and 7, respectively.

## 3. Results and discussion

Electrical resistivity of the samples sintered at  $830^\circ\text{C}$  showed a semiconducting behavior. The resistivity of the samples sintered at  $840^\circ\text{C}$  decreases slowly with decreasing temperature, however a complete disappearance of resistance has not been observed, as shown in Fig. 1a.

Samples prepared at  $850^\circ\text{C}$  exhibit a superconducting

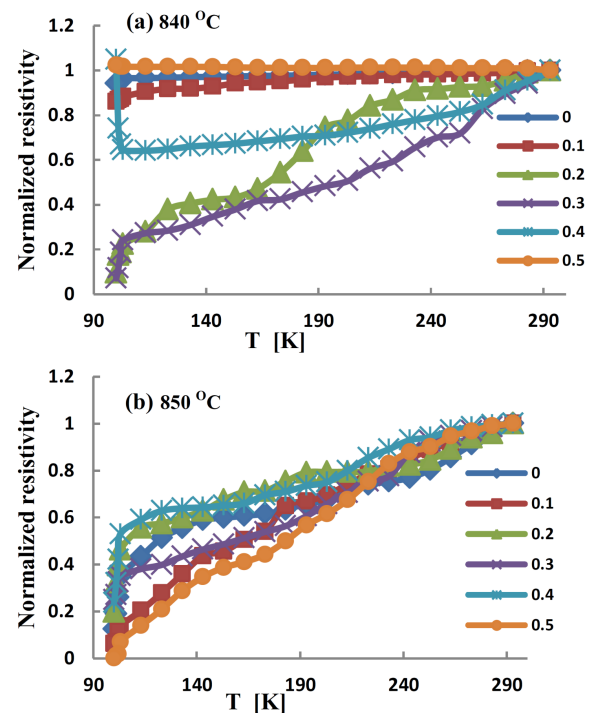


Fig. 1. Temperature dependence of resistance of the samples prepared at (a)  $840^\circ\text{C}$  and (b)  $850^\circ\text{C}$ .

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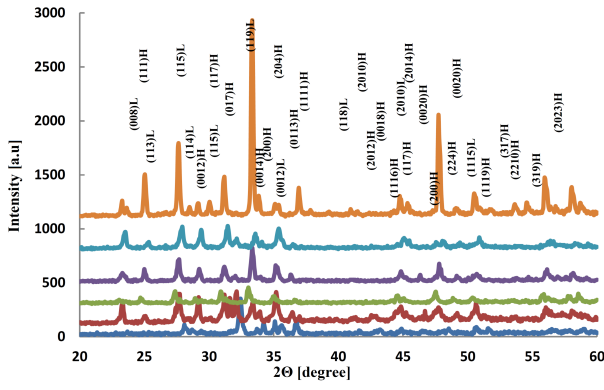


Fig. 2. X-ray diffraction patterns of the samples.

behavior, as shown in Fig. 1b.  $T_C$  was found to be 115, 104.5, 105, 109, 115 and 128 K for  $x = 0, 0.1, 0.2, 0.3, 0.4$  and  $0.5$ , respectively. The  $T_C$  has been increasing with increasing K concentration for  $x = 0.4$  and  $0.5$ . This could be assigned to the presence of K, which increases the amount of impurities and decreases the melting temperature of the samples.

Impurities can act as the pinning centers to fix vortices or they may enhance connectivity among the grains during the sintering process. A highest transport was observed at  $x = 0.5$ , which means that

a higher resonant tunneling had occurred between  $\text{CuO}_2$  layers through localized centers along the  $c$ -axis.

There was a negative result for the samples produced at higher sintering temperature of  $860^\circ\text{C}$ . XRD pattern of the samples sintered at  $850^\circ\text{C}$ , presented in Fig. 2, shows orthorhombic structure with two main phases, high- $T_C$  phase (2223) and low- $T_C$  phase (2212). The most intense peak pattern belongs to the high- $T_C$  phase due to the enhanced grain growth and better orientation of grains with K substitution. The relative volume fractions (VF, [%]) of the Bi-2223 (HP) and Bi-2212 (LP) phases were estimated using equation from [2]. The results are listed in Table I. It can be observed that the fraction of the high- $T_C$  phase 2223 and the  $c$  parameter have increased beyond  $x = 0.3$ .

TABLE I  
Values of  $a$ ,  $b$ ,  $c$ ,  $V$ , and VF of the samples.

$x$	$a$ [Å]	$b$ [Å]	$c$ [Å]	$V$ [Å <sup>3</sup> ]	VF [%]	
					HP	LP
0.0	5.415	5.4510	37.727	1113.97	80.93	19.06
0.1	5.407	5.3453	37.215	1075.62	63.97	36.02
0.2	5.352	5.5430	37.350	1108.19	59.30	37.23
0.3	5.441	5.4401	37.077	1097.54	57.08	39.36
0.4	5.421	5.4232	37.263	1095.56	69.08	27.90
0.5	5.370	5.4690	37.487	1100.99	64.66	31.47

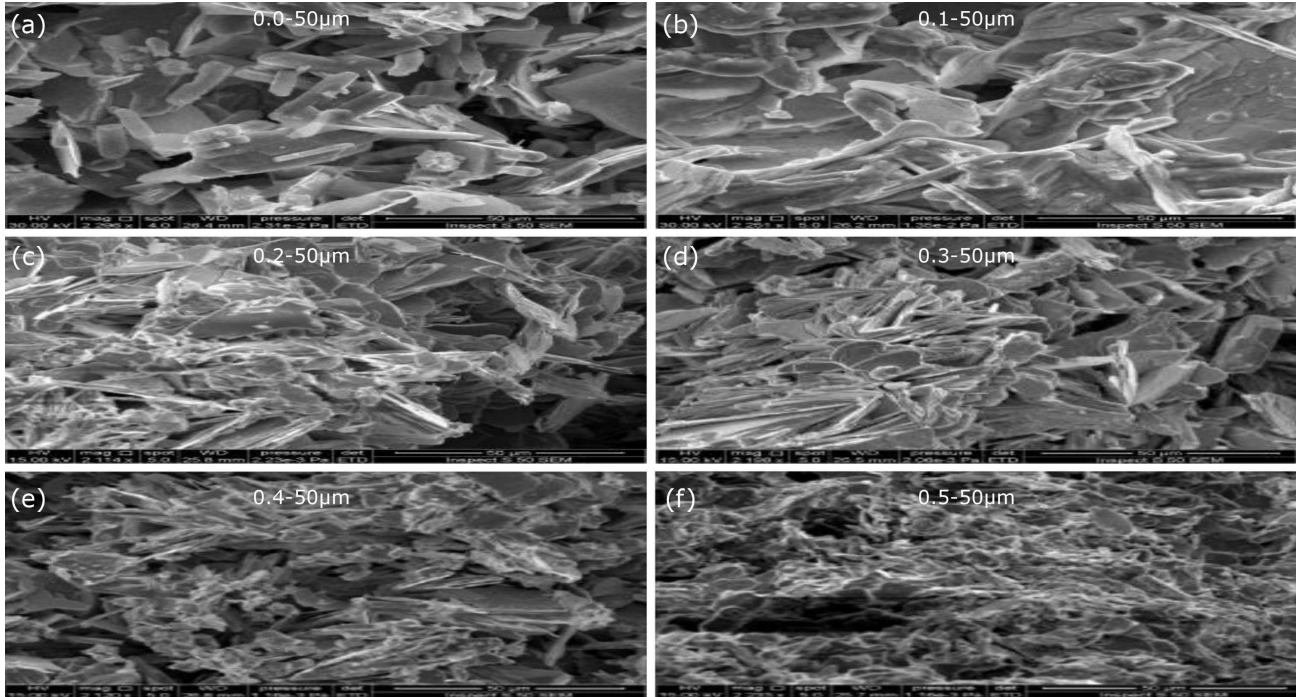


Fig. 3. SEM micrographs of the samples.

The SEM images of undoped sample show cylindrical-like crystals with irregular sizes and stacks of plate-like structure, as shown in Fig. 3a. Each of these stacks is composed of thousands of layers. Some of the plates

have grown one through the other, giving rise to bigger grains. Doping with K at  $x = 0.1$ , as presented in Fig. 3b, results in thin plate and flake-like crystals with no preferred orientation and some precipitation of par-

ticles on the surface. Samples with  $x = 0.2$  and  $0.3$ , as shown in Fig. 3c and d, respectively, show discontinuous /porous microstructure packets growing in random directions and voids produced between these packets. Each clump contains a group of plate-like crystals oriented randomly. On the other side, concentration of  $0.3$  decreases the grain size, voids and porosities. Increasing  $x$  to  $0.4$  and  $0.5$ , as shown in Fig. 3e and f, respectively, increases the contact area between grains, and yields a smoother and denser surface. In our opinion, existence of K at the grain boundaries leads to coalescence of the grains.

A series of hardness measurements were done on the samples sintered at  $850^\circ\text{C}$  and the results are presented in Figs. 4 and 5. It is apparent from Fig. 4a that the microhardness values were decreasing non-linearly as the applied load increased until about  $0.96\text{ N}$ , a threshold above which Hv values tend to attain saturation. This type of non-linear behavior is known as indentation size effect [5]. This behavior can be interpreted as follows. At larger indentation loads, the Hv registered smaller values. This observation may be due to the presence of impurity phases and irregular grain orientation. At smaller indentation loads, the Hv recorded higher values, which is ascribed to the fact that the measured hardness values were more indicative of the monocrystalline state without interference from the grain boundaries.

The variation of microhardness Hv with increasing K content at certain applied loads is shown in Fig. 4b. On

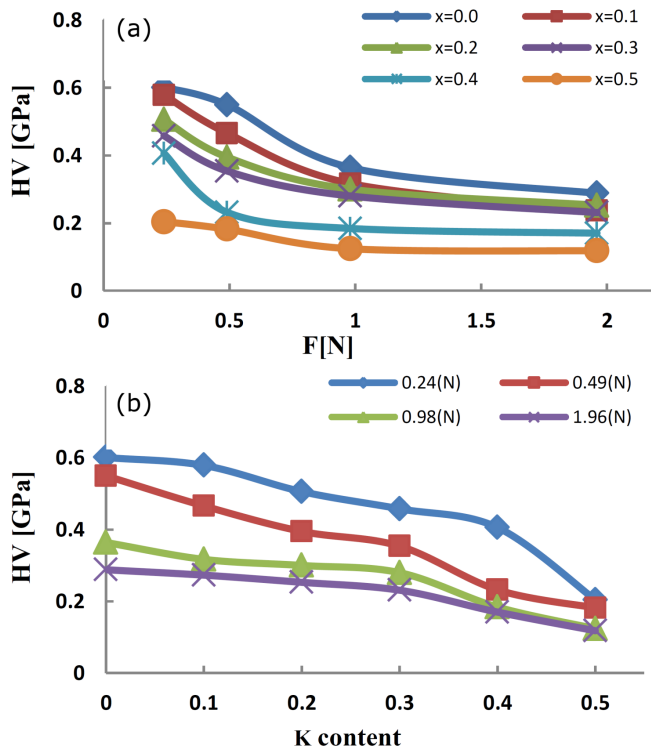


Fig. 4. Variation of Hv of the samples as a function of (a) applied load and (b) K content.

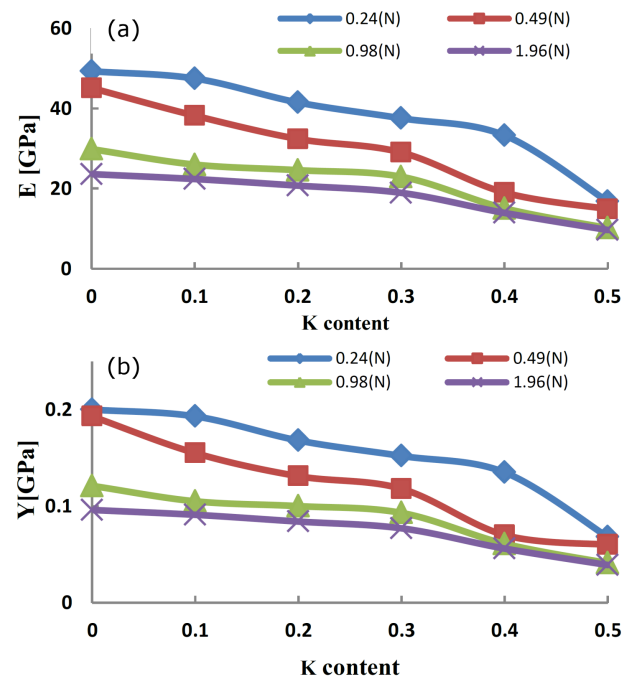


Fig. 5. Variation of (a)  $E$  and (b)  $Y$  of the samples as a function of K content.

the other side, substitution with K changes the surface micrographs of the samples. This would produce many grain boundaries in the structure, and consequently the mechanical properties of the samples would be affected differently.

$E$  and  $Y$  decreased gradually with increasing K content, as shown in Figs. 5a and b, respectively. This decrease is attributed to the formation of impurity phases and irregularities, mainly distributed at the grain boundaries, which lead to distortion of the bond strength, and consequently the hardness values decrease. Brittle fracture can occur in an intergranular manner when the crack propagates along grain boundaries.

The increase of the K concentration causes two effects: substitution of lattice atoms, when K atoms become part of the structure, as well as enrichment and uniform distribution on the grain boundaries in great amounts, which might be one of the causes of brittleness. Mechanical properties can be explained in terms of resistance to dislocation motion and bonding force between the atoms. The general contribution to the resistance is mainly of two types: (i) the intrinsic resistance and (ii) the resistance due to imperfections. In our case, decrease in the Hv,  $E$  and  $Y$  of the samples with increasing K substitution may be due to both, the pinning of dislocations at the impurity sites and other defects caused by the presence of impurity atoms in the crystal, and the variation in the magnitude of the bond forces in the crystal containing impurities. Samples with high doping  $x = 0.5$  had fractured with increasing applied load.

#### 4. Conclusions

The optimum sintering temperature for studied samples appears to be 850 °C. It did improve the superconductivity properties and increased the amount of the high- $T_C$  phase. The substitution by K improves the crystalline arrangement degree and volume fraction of the 2223 phase. Samples with K substitution of  $x = 0.5$  exhibited the largest value of  $T_C = 128$  K. Mechanical properties of the samples were found to be load dependent. The results revealed that the substitution of K leads to brittleness of the samples, which causes deterioration of the mechanical properties.

#### References

- [1] M.M. Abbas, L.K. Abbas, U. Salman, *Ener. Procedia* **18**, 215 (2012).
- [2] M.M. Abbas, S.F. Oboudi, N.Q. Raoof, *Mater. Sci. Appl.* **6**, 55420 (2015).
- [3] B. Özkurt, *J. Supercond. Nov. Magn.* **26**, 261 (2013).
- [4] U. Kölemen, O. Uzun, C. Emeksiz, B. Özçelik *J. Supercond. Nov. Magn.* **26**, 3215 (2013).
- [5] I.J. McColm, *Ceramic Hardness*, Plenum, New York 1990.
- [6] H.A. Cetinkara, M. Yilmazlar, O. Ozturk, M. Nursoy, C. Terzioglu, *Intern. IOP Publishing J. Phys. Conf. Series* **153**, 012038 (2009).
- [7] M. Yilmazlar, O. Ozturk, H. Aydin, M. Akdogan, C. Terzioglu, *Chinese J. Phys.* **45**, 128 (2007).