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Research Article

Mechanical Properties of $Bi_{2-x}Cd_xPb_{0.3}Sr_2Ca_2Cu_3O_{10+\delta}$ superconductors

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Abstract

Solid state reaction methods were used to prepare a high temperature superconductor with a nominal composition Bi2−xCdxPb0.3Sr2Ca2Cu3O10+δ for (0≤x≤ 0.5). The prepared samples have been investigated through X- ray diffraction measurements to determine structural properties. Superconducting properties by dc electrical resistivity and mechanical properties by static Vickers hardness measurements have been carried out to assess the effects of Cd substitution. X-ray diffraction analysis showed two phases: high-TC phase 2223 and low-TC phase 2212 with orthorhombic structure for all samples. The optimum concentration was found for 0.2 which improved the microstructure and had the highest TC value 126 K for maximum ratio of c/a. Cd doping increased Vickers hardness, Young's modulus and yield strength values. Load dependent values of Hv, E, and Y are greater than those of the load independent values. Possible reasons for the observed improvements in the superconducting and mechanical properties due to Cd substitution are discussed.

Keywords: *The author can include 5-7 words like Thermal Analysis, Pre-conditioner, In-mold, Inoculant's efficiency.*

1. Introduction

Bi-based compounds are considered to be the most promising material for the application of high-T_C superconductors. There are three superconducting phases in the BiSrCaCuO system which have an ideal structural formula $Bi_2Sr_2Ca_{n-1}Cu_nO_{4+2n}$, with n = 1, 2 or 3 indicating the number of the CuO2 planes. Synthesis of a nearly pure single-phase high-TC 2223 phase (TC≈110 K) is a complicated process due to its extremely narrow stability range [Margiani and *et al* 2012]. On the other hand Bi-2212 transformation into Bi-2223 phase, is very slow and cannot be completed.
The formation and stability of the 2223 phase can be modified by addition or substitution a varying ionic radii elements and bonding characteristics. This variation is thought to be related to the density of charge carriers in the CuO planes [Takano *et al* 1988]. Pb doping was found to reduce the partial melting temperature and produced more liquid phase during sintering, which in turn improved the rate formation of Bi-2223 phase [Mahmood and *et al* 2012].

Researchers observed that Cd substitution for Bi improved the superconducting properties of the Bi-Pb-Sr-Ca-Cu-O system and remarkably enhances the Bi-2223 phase formation and stabilization [Ghahfarokhi and *et al* 2010; Shoushtari and *et al* 2011].

The determination and development of the Bi-2223 compounds mechanical properties is as important as superconducting parameters in choosing the application field of these high-temperature superconductors [ZAN and *et al* 2009; Ersin and *et al* 2015; Aydin *et al* 2009].

Accordingly, a number of studies have been carried out recently to improve the mechanical properties of superconductors [Özkurt 2013; Kölemen and *et al* 2013].

Hardness is the measure of the resistance of a material against a load applied to its surface. Vickers microhardness testing is a convenient method to investigate the mechanical properties so it is frequently used to solids in the form of bulk samples. If the deformation is due to very small applied loads, elastic deformation is observed. Forces above some critical value produce plastic (irreversible) deformation. It is well known that the microhardness of solids depends on the applied load. Generally, the microhardness value decrease with increase in the applied load, which is known as ISE (Indentation Size Effect) [Gong and *et al* 1999 ;Elmustafa and *et al* 2003]. Another case is RISE (Reverse Indentation Size Effect) [Khalil 2003; Sangwal 2000], where the microhardness increases with applied load.

The objective of this paper is to investigate the role of Cd dopant and its influence on the mechanical properties such as hardness, elastic modulus, and yield strength as well as the critical temperature of $Bi_{2-x}Cd_xPb_{0.3}Sr_2Ca_2Cu_3O_{10+\delta}$ samples with different amounts of Cd concentration.

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2. Experiment

The samples of the system $Bi_{2-x}Cd_xPb_{0.3}Sr_2Ca_2Cu_3O_{10+\delta}$ with concentration ($0 \le x \le 0.5$) were prepared by conventional solid-state reaction route. An appropriate weights of the starting materials, Bi2O3, PbO, Sr(NO3)2,CaCO3, Cu2O and CdO were taken with precise values. Then the powders were well mixed and grounded using agate mortar with a sufficient quantity of 2-propane to homogenize the mixture to get a fine powder. The mixture was then calcined in air at 800˚C for 24 hrs. The powder obtained reground again and pressed into disc-shaped pellets at 0.7 GPa, with 13 mm diameter using hydraulic pressure type (Specac).The pellets were sintered in air atmosphere at 840˚C for 140 hrs

The structure of the prepared samples was obtained by using X-ray diffractometer type Philips with the Cu-Kα radiation. A computer program was used to calculate the lattice parameters, based on Cohen's last square method.

The resistivity measurements were performed by the standard four-probe method.

Hardness measurements of the samples were performed on the polished surface of the examined samples with a digital micro-hardness tester type (HVS-1000) at room temperature. The applied load (F) varied in the range 0.245–2.940 N and applied for 15 seconds. The indenter was pressed on the polished different surfaces of the samples making sure that the indentations do not overlap.

The load dependent (Vickers) microhardness values of the samples are calculated using the relation [McColm 1990]:

$$Hv = 1854.4\ (F/d2) \quad (1)$$

Where d is the diagonal length of the indentation mark in μm.

The Young modulus E of superconductors is related to the Vickers microhardness by the relation [16 Cetinkara and *et al* 2007]:

$$E = 81.96\ Hv \quad (2)$$

The yield strength Y is related to the hardness by the relation [Ylimizer and *et al* 2009]:

$$Y = Hv / 3 \quad (3)$$

3. Results and Discussion

XRD analyses showed an orthorhombic structure of all the samples and show two main phases: high-T_C phase (2223), low-TC phase (2212) with some impurities phases like CuO detected at 2θ around 37^0 as shown in Figure 1. The appearance of more than two phases could be related to the stacking faults along the c-axis.

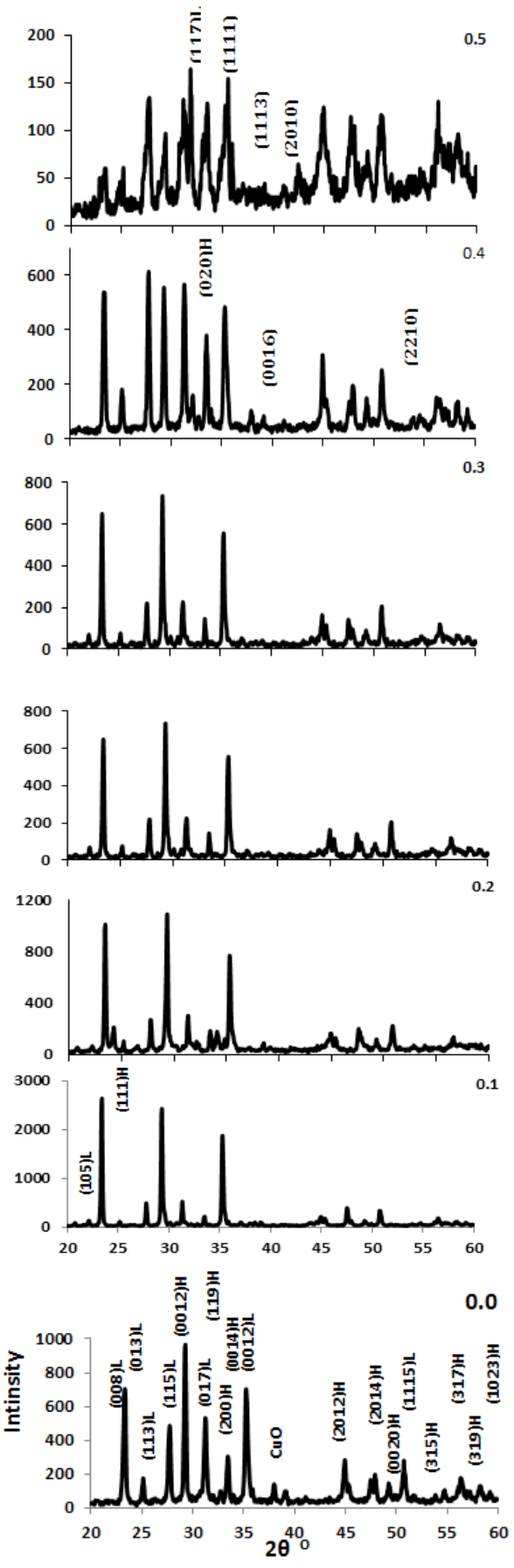

Fig. 1 XRD pattern of $Bi_{2-x}Cd_xPb_{0.3}Sr_2Ca_2Cu_3O_{10+\delta}$ compounds

According to the model suggested by Grivel and Fliikiger [Gundakaram and *et al* 2001,] the (Bi,Pb)-2223 phase forms not due to a layer-by-layer intercalation in the pre-existing (Bi,Pb)-2212 grains but through a distinct nucleation and growth process.
It should be noted that the relative intensity of the diffraction peaks vary with different samples while peak positions varies slightly with increasing the Cd concentration likewise, resulted, a change in the lattice constant of the sample. Interestingly, samples with x =0.1- 0.2 showed intense peaks pattern due to the enhanced grain growth and better orientation of grains with Cd diffusion.

The parameters a, b, c , V and c/a were calculated and listed in Table 1. As the Table shows c parameter decreases monotonously with the Cd concentration. Moreover, upon Cd substitution a parabolic curve for c/a ratio is observed as illustrated in Figure 3, that is increased up to x = 0.2 then shortened towards Cd concentration. This implies that the mechanism of substitution of Bi by Cd is not simple. This result may be related to the substitution which causes increasing oxygen content in the unit cell. Moeover, since the ionic radius of Bi^{+3} ($1.03A^0$) is greater than the ionic radii of Cd^{+2} ($0.95A^0$) thus the substitution decrease both a and c parameters which leads to increases the distance between the CuO2 planes. But increasing Cd concentration causes reduction of the c parameter while the a parameter increases. This could be ascribed to the charge ordering phenomenon (probably induced as a pair breaker) may be a companied by change in oxygen content or oxygen ordering effects. Furthermore, the interaction between additional bands crosses the Fermi level extracts the holes from CuO band [Azhan and *et al* 2009]. This attractive interaction caused the decreased of the distance between CuO2 planes.

On the other hand the addition of Pb to the compounds may relax the modulation by influencing the charge balance, oxygen content and structural of the relevant layers [lkeda and *et al*. 1988].

Table 1 Lattice parameters of Bi2−x CdxPb0.3Sr2 Ca2 Cu3O10+δ compounds with different concentrations

x	a	b	c	V
0	5.451443	5.45248	37.45907	1113.429
0.1	5.398889	5.471745	37.32115	1102.517
0.2	5.381182	5.459116	37.23884	1093.947
0.3	5.420641	5.454968	37.24647	1101.357
0.4	5.44252	5.45223	37.10309	1100.992
0.5	5.442769	5.562284	37.04279	1121.442

Measurements of electrical resistivity of the samples are displayed in Figure 4. It is found that increasing Cd concentration possess somewhat lower resistivity, metallic behavior in the normal state and superconducting transition to zero resistance with T_C = (111.5, 121.5 and 126.5) K for (x=0.0, 0.1 and 0.2) respectively. Enhancement of Tc with increasing Cd could mainly due to increasing amounts of Bi-2223 phase the reason may be attributed to the existence of the high Tc- phase as referred in the x-ray analysis, as well as to the strong link and increasing the contact areas between the grains during the sintering process in other words decrease of porosity [Heh 1990]. Whereas, beyond 0.2 resistivity of the samples in the normal state increased with increasing Cd concentration. Moreover, the samples showed semiconductor behavior before superconductor transition to zero with T_C = (107 and 105) K for (x=0.3 and 0.4) respectively. While the sample for the composition with x=0.5 is behaved non-metalic. This could be attributed to the substantial degradation of the (Bi,Pb)-2223.

It is well established that there are two types of superconducting grains, one formed by the 2223 phase and the second by the 2212 phase coupled together via some weak links and by passes the islands of the 2212 phase [Bolat and *et al* 2000].

Once the volume fraction of 2223 phase within the sample is sufficient to make this possible, a one-step resistivity transition is observed even in the samples which contain a rather large amount of the 2221 phase. Most of hole-doped cuprates (but not in all) has bell-like shape for T_C as a function of doping which is common behavior in mono-or bilayer cuprates. Consequently, the different doping regions of the superconducting phase may be chosen such as the underdoped, optimally doped and overdoped regions [Fujii and *et al* 2002 ; Parinov 2012].

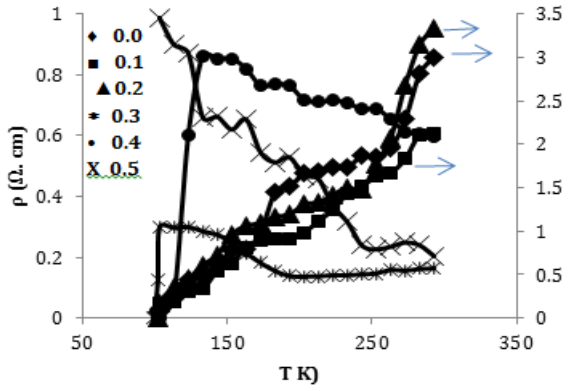

Fig. 2 Temperature dependence of resistivity for Bi2−x CdxPb0.3Sr2Ca2Cu3O10+δ compounds with different concentrations

According to the above results the relation between T_C and Cd concentration is almost parabolic as given in Figure 3, and can be explained based on the parabolic dependence between TC and the number of holes per CuO2 layer [Mihalache and *et al* 2006]. This result in good agreement with that obtained by [Abbas and *et al* 2015].

Highest T_C was determined at 0.2 actually appears for a sufficiently large c/a ratio, i.e. when the 2D

character is strong enough, which confirm that the sample is in optimal doping regime while decrease of T_C beyond this concentration seems to be due to the shift of this sample towards the over-doped region.

From the viewpoint it is obviously each of T_C, and c/a parameter has harmonious behavior with doping concentration.

Table 2 Experimental procedure parameters

x	c/a	T_C
0	6.871405	111.5
0.1	6.912746	121.5
0.2	6.920197	126.5
0.3	6.87123	107
0.4	6.817263	105
0.5	6.805872	-

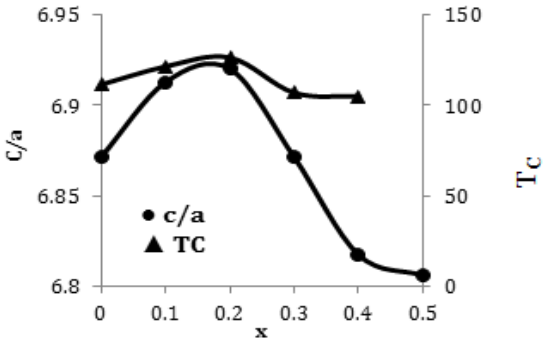

Fig. 3 Variation of T_C and c/a ratio with different Cd concentrations.

The Vickers micohardness Hv, yield strength Y and Young's elastic modulus E results calculated are presented in Table 3. It is clear from the table that the Hv values for all the samples depend strongly on the applied loads and Cd substitution. The variation of the Hv with regard to the applied loads, on the surfaces of the samples is exhibited in Figure 5. it is observed that the load dependent microhardness values of samples 0.0-0.3 descends nonlinearly as the applied load increases until, at about 0.96N, a threshold above which Hv values tend to attain saturation, demonstrated the ISE behavior. This behavior can be interpreted as follows; at larger indention loads, the Vickers hardness registered smaller values, this observation may be due to the presence of impurity phases and irregular grain orientation distribution.

While smaller indention loads, the Vickers hardness recorded higher values, this is ascribed to the fact that measured hardness values were more indicative of the monocrystalline state without interference from grain boundaries, [Yimazlar and *et al* 2006]. Similar changes in the Vickers hardness and phases were reported by [Khalil 2001].

Table 3 Hv, Y and E values with different concentrations of Bi2−x CdxPb0.3Sr2Ca2Cu3O10+δ

x	F(N)	Hv (GP)	Y(GP)	E (GP)
0.0	0.245	1.218179	99.84199	0.40606
	0.49	1.048166	85.90771	0.349389
	0.98	0.534379	43.79774	0.178126
	1.96	0.476726	39.07246	0.158909
	2.94	0.326587	26.76709	0.108862
	4.9	0.452509	37.08765	0.150836
0.1	0.245	0.285626	0.285626	0.285626
	0.49	0.206977	0.206977	0.206977
	0.98	0.139543	0.139543	0.139543
	1.96	0.110265	0.110265	0.110265
	2.94	0.095753	0.095753	0.095753
	4.9	0.144267	0.144267	0.144267
0.2	0.245	0.741524	60.77533	0.247175
	0.49	0.648694	53.16698	0.216231
	0.98	0.589861	48.34502	0.19662
	1.96	0.395284	32.39752	0.131761
	2.94	0.305565	25.04414	0.101855
	4.9	0.478765	39.2396	0.159588
0.3	0.245	0.681866	55.88574	0.227289
	0.49	0.657526	53.8908	0.219175
	0.98	0.652966	53.51713	0.217655
	1.96	0.369343	30.27136	0.123114
	2.94	0.333702	27.35018	0.111234
	4.9	0.705203	57.79843	0.235068
0.4	0.245	0.76861	62.99526	0.256203
	0.49	0.606888	49.74052	0.202296
	0.98	0.343864	28.18308	0.114621
	1.96	1.293642	106.0269	0.431214
	2.94	1.373333	112.5584	0.457778
	4.9	1.539821	126.2037	0.513274

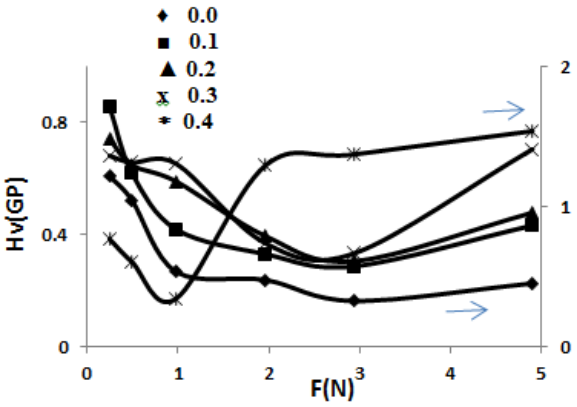

Fig. 4 Variation of Hv as a function of applied load

The Hv for Sample 0.4 descends as the load increases up to 0.96N. This observation is associated with the weak grain boundaries of the sample. For higher applied loads Hv increases exhibit RISE and converge

to a plateau region. The Hv values at the plateau region are considered to be the load independent real hardness values. This could be due to specimen cracking. Excessive cracking in the sample decreases the elasticity and thus Hv value of the sample, thereafter, no elastic recovery, but only plastic deformation is observed [Cavda and *et al* 2012 ; Dogruer and *et al* 2014] .

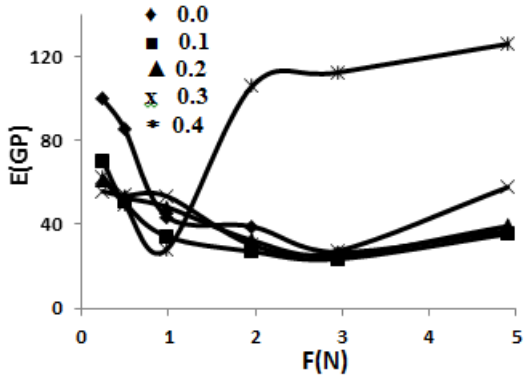

Fig.5 Variation of E as a function of applied load

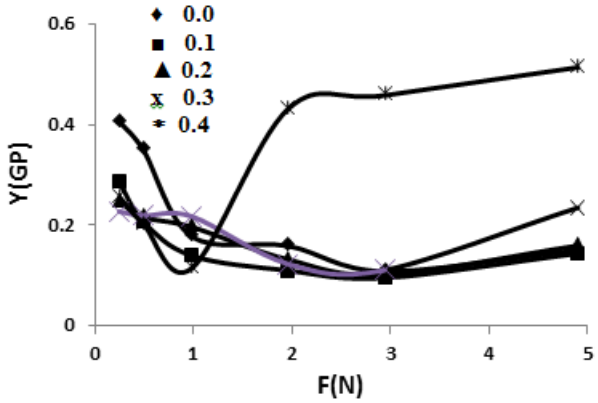

Fig. 6 Variation of Y as a function of applied load

The variation of Hv, Y, and E with increasing Cd-content at certain applied loads of the superconducting samples is shown in Figures (7,8 and 9) respectively.

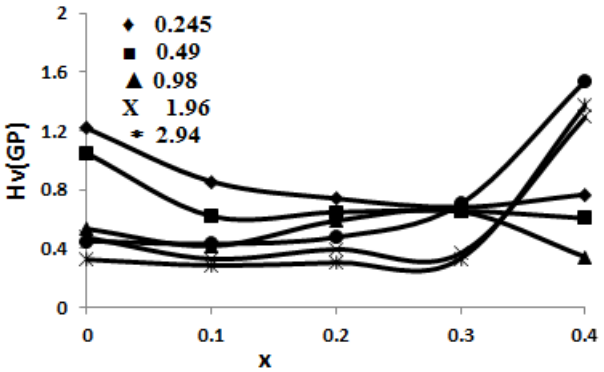

Fig. 7 Variation of Hv with different Cd concentrations.

These Figures demonstrate that Hv, E and Y increased gradually with increasing Cd content. This may be ascribed to amount of doping in samples filling the intergrain space resulting in better grain growth and causing larger grains which leads to improve the strength of connection between superconducting grains as indicated by [Jannah and *et al* 2009].

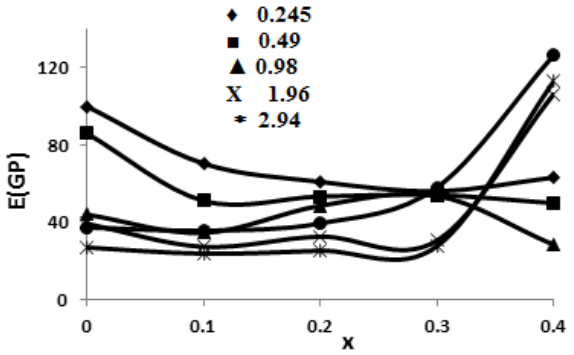

Fig. 8 Variation of E with different Cd concentrations.

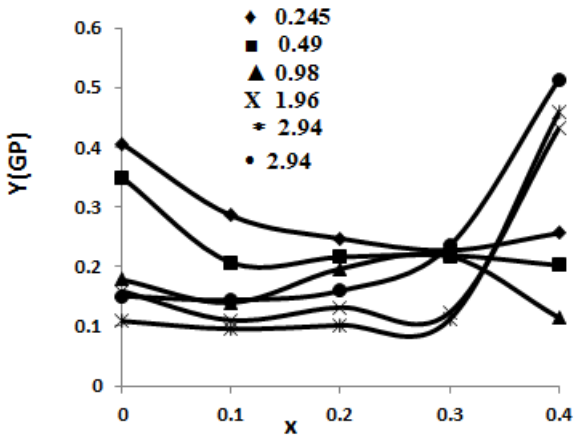

Fig. 9 Variation of Y with different Cd concentrations.

Conclusions

Substituted Bi partially by Cd in Bi2−xCdxPb0.3Sr2Ca2Cu3O10+δ system, promote the 2223 phase of samples and showed that the maximum Tc = 126K at the concentration 0.2 in optimal doping regime. The improvement of the superconducting properties of samples is due to the modification of the grain boundaries together with better crystallinity and larger grains The XRD and electrical resistivity results of samples indicating that the variation in lattice parameters and superconductivity behavior are mainly due to the structure distortion and charge ordering phenomenon induced by Cd substitution in Bi sites.

Moreover, increasing Cd concentration strengths interlayer bonding as confirmed by the increase in the Hv, E and Y values of Bi2−xCdxPb0.3Sr2Ca2Cu3O10+δ. ISE behavior is observed for the samples with Cd concentration 0.0-0.3. While, the reverse type of indentation size effect (RISE), was obtained for concentration 0.4 and revealed to the indentation-induced specimen crack.

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