Düzce University
Journal of Science & Technology

Research Article

The Fracture Toughness and Brittleness of Au doped YBCO Superconductors

Rıfkı TERZİOĞLU [a*]

[a] *Electrical Electronics Engineering Department, Bolu Abant Izzet Baysal University, Bolu, TURKEY*
**Corresponding author E-mail adress: rifkiterzioglu@ibu.edu.tr*
DOI: 10.29130/dubited.652403

ABSTRACT

In this work, the effect of gold addition on fracture toughness and brittleness of $YBa_2Cu_3O_{7-x}$ (YBCO) superconductors were analyzed. Five different samples (undoped, 1%, 5%, 15% and 20%) were produced using the conventional solid-state reaction (SSR) method. The experimental results of the microhardness measurements are used to obtain the yield strength, Young's modules, fracture toughness and brittleness index. Some of these mechanical properties mentioned above are extracted by using the proportional specimen resistance (PSR) model. The results showed that the fracture toughness and brittleness of the samples were load dependent. The yield strength, Young's modules, brittleness index and fracture toughness values increase with decreasing test load and decreases with increasing gold content. The possible reasons of the alteration in the mechanical properties due to gold addition are analyzed.

Keywords: *Au addition, Fracture toughness, YBCO*

Au Katkılı YBCO Süperiletkenlerin Kırılma Tokluğu ve Kırılganlıkları

ÖZET

Bu çalışmada, altın ilavesinin YBCO süperiletkenlerinin bazı mekanik özelliklerine etkisi incelenmiştir. Geleneksel katı hal reaksiyon metodu kullanılarak beş farklı numune (katkısız, % 1, % 5, % 15 ve % 20) hazırlanmıştır. Mikro sertlik ölçümlerinin deneysel sonuçları, akma dayanımı, Young modülleri, kırılma tokluğu ve kırılganlık indeksi elde etmek için kullanılmıştır. Yukarıda belirtilen bu mekanik özelliklerin bazıları PSR modeli kullanılarak elde edilmiştir. Numunelerin mekanik özelliklerinin yüke bağlı olduğu bulunmuştur. Akma dayanımı, Young modülleri, kırılganlık indeksi ve kırılma tokluğu değerlerinin azalan test yükü ile arttığı ve artan altın içeriği ile azaldığı gözlemlenmiştir. altın ilavesi nedeniyle mekanik özelliklerde gözlenen bozulmanın olası nedenleri tartışılmıştır.

Anahtar Kelimeler: *Au katkılama, Kırılma tokluğu, YBCO*

Geliş: 28/11/2019, Düzeltme: 02/03/2020, Kabul: 04/03/2020

I. INTRODUCTION

High T_c superconductors (HTS) are the most ambitious candidates for industrial applications and technical advances have been achieved in recent years [1-4]. Especially remarkable progress in YBCO coated conductors (YBCO CC) has been achieved. YBCO CC's have high J_c and good mechanical properties which make them useful superconductors. Current research destination in HTS's are higher J_c and better mechanical properties.

Diffusion, chemical doping and addition have a strong effect in cuprate superconductors. Many studies have been done to improve the superconducting, electrical, magnetic and mechanical properties of superconductors [5-13]. In Ref. [10] the structural and electrical properties of Lu-doped YBCO superconductors were analyzed. They have found that the transition temperature and the activation energy decrease with Lu-doping. YBCO and BSCCO are the most used superconductors in industrial applications so improving the electrical and mechanical properties of these materials have been the goal in studies [14,15].

Wires and tapes are generally used in superconductors engineering applications. The mechanical properties of such superconductors are crucial. Therefore, beside good superconducting properties, superconductors need to have good mechanical properties like being strong and flexible. Fracture toughness, yield strength and hardness are important mechanical parameters and many researches have been made to understand these parameters and behaviors [16-20]. The influence of diffusion-annealing temperature on the mechanical properties of gold doped Bi-223 superconductors were analyzed [17]. Mechanical properties of the samples were found to be load dependent and explained with the typical indentation size effect (ISE). In another study [20] the Vickers hardness properties of YBCO bulk superconductors at cryogenic temperatures were analyzed. It was reports that the Vickers hardness and indentation scars increased with decreasing temperature.

In previous work [15] Au doped YBCO samples were investigated and the research consists of scanning electron microscopy (SEM), X-ray diffraction (XRD), DC resistivity, alternative current magnetic susceptibility and energy dispersive X-ray fluorescent (EDXRF) measurements. The increasing addition of gold in YBCO from x=1wt% to x=20wt% decreased the T_c and J_c values.

The results of microhardness measurements are used to investigate the effect of Au addition on the yield strength, Young's modules, brittleness index and fracture toughness which are calculated analytically.

II. EXPERIMENTAL DETAILS

The microhardness measurements can be done in different methods. In this study, the most common method which is the Vickers microhardness test method is used. Vickers microhardness measurements were performed at room temperature and the applied test load was applied for 10 seconds. In this study the applied load values were 0.245, 0.49, 0.98, 1.96 and 2.940 N. Indentations were not overlaped for accurate results. For this reason, the indenter was contacted on different parts of the sample and the Vickers microhardness values were obtained and analyzed by this way. The gold added YBCO bulk samples were prepared by the standard SSR method.

The samples were prepared with Y_2O_3, $BaCO_3$ and CuO. The powders were weighted with a precise microbalance and mixed by a pestle for 6 hours. The calcining process was done at 930℃ for 24 hours. The calcined powder was ground and remixed powder shaped by using a suitable die. After this process they were pressed under 230 MPa to give the desired shapes. Sintering was done in 930℃ and in an O_2 atmosphere. The samples were cooled in an atmosphere of flowing O_2. More detail of sample preparation can be found in previous study [15]. Sample numbers and the amount of gold addition are given in Table 1.

Table 1. *Sample properties*

Sample	*Au addition (% w.t.)*
Y0	Undoped
Y1	1
Y2	5
Y3	15
Y4	20

III. RESULTS AND DISCUSSION

The microhardness values were found to be test load and Au doping dependent. The dependency was explained with the reverse indentation size effect (RISE).

Table 2. *Vickers microhardness measurement [21]*

Samples	*F (N)*	*d (μm)*	*H_v (GPa)*
Y0	0.245	13.62	2.448
	0.49	16.7	3.258
	0.98	21.97	3.765
	1.96	27.55	4.786
	2.94	33.62	4.823
Y1	0.245	14.28	2.226
	0.49	18.67	2.604
	0.98	23.37	3.325
	1.96	31.46	3.671
	2.94	38.32	3.711
Y2	0.245	16.83	1.602
	0.49	20.22	2.221
	0.98	25.97	2.694
	1.96	33.94	3.153
	2.94	40.9	3.258
Y3	0.245	19.83	1.155
	0.49	24.14	1.558
	0.98	29.8	2.045
	1.96	40.63	2.201
	2.94	47.73	2.392
Y4	0.245	22.91	0.865
	0.49	29.66	1.032
	0.98	32.57	1.713
	1.96	42.2	2.04
	2.94	49.3	2.242

The Vickers microhardness values, H_v, were calculated for different applied loads by using the equation below [22],

$$H_v = 1854.4(F/d^2) \quad (1)$$

where F is the applied load in N and d is the diagonal length of the indentation mark in µm's. Diagonal lengths, applied loads and calculated hardness values using Eq. (1) have been presented here in Table 2 to discuss and calculate mechanical properties such as fracture toughness and brittleness index in this study.

Elastic modulus/Young's modulus (E) is the tensile elasticity of a material and yield strength is the stress at which a material begins to plastically deform. The elastic modulus-hardness relation is shown in Eq. 2 [23],

$$E = 81.9635H_v \quad (2)$$

The yield strength, is calculated by the relation [24, 25],

$$Y \approx H_v/3 \quad (3)$$

Fracture toughness (K_{IC}) is the ability of a material to resist fracture. K_{IC} which is the critical stress intensity factor is directly related to γ (the surface energy) of the crack faces is given by [26],

$$K_{IC} = \sqrt{2E\gamma} \quad (4)$$

In [27] and [28] the ISE behavior is shown by Eq. (5),

$$F/d = H_0 d + \gamma \quad (5)$$

where γ denotes the surface energy and H_0 is load independent microhardness constant. Further, the H_{PSR} value can be inferred from the PSR model by way of the formula,

$$H_{PRS} = 1854.4H_0 \quad (6)$$

The *F/d* against *d* graph along with the linearly regressed PSR model (Eq. (5)) gives the true hardness and surface energy. The slope of the graph is the true hardness, H_0, and the intercept of each line represents the surface energy, γ. The extracted values of H_0, γ, *LRC* and H_{PRS} calculated by Eq. (6) for samples are listed in Table 3 [21].

Table 3. *H_0, γ, R and H_{PRS} values for samples [21]*

	H_0 (GPa)	γ (N/µm)	*R*	H_{PSR} (GPa)	H_v in plateau region
Y0	0.00355	-0.0305	0.989	6.583	4.786-4.823
Y1	0.00254	-0.0192	0.994	4.710	3.671-3.711
Y2	0.00239	-0.0245	0.998	4.432	3.153-3.258
Y3	0.00193	-0.0211	0.994	3.579	2.201-2.392
Y4	0.00173	-0.0356	0.969	3.208	2.04-2.2421

The values of γ that are shown in Table 3 are negative which is an expected result that show RISE behavior. This means that there is no elastic deformation, but plastic deformation is observed [21].

Another finding is that the H_0 parameter decreased while the γ parameter changed randomly with increasing Au addition. It is seen from Table 3 that the true hardness value calculated from the PSR model (4.432 G Pa) of Y2 is higher than the value in the plateau region (H_v =3.153-3.258 GPa) [21]. This value is same for all samples. So, the hardness values calculated from the PSR model for our data lies beyond the plateau region.

Also, the brittleness index (the ratio of uniaxial compressive strength to tensile strength), B_i, can be calculated using the relation [29],

$$B_i = H_v / K_{IC} \tag{7}$$

The values of load dependent mechanical properties were calculated for each load by using Eqs. (2, 3, 4 and 7), and they are summarized in Table 4 (E and Y) and Table 5 (K_{IC} and B_i) for all the samples.

As seen in Table 4, increasing loads causes an increase in the E and Y values. The effect of adding gold to samples decrease the E and Y values. This degradation is explained by the increasing voids, impurity phases and modification of the grain boundaries [15].

Table 4. *E and Y values of samples*

Sample	*F* *(N)*	*E* *(GPa)*	*Y* *(GPa)*
Y0	0.245	200.647	0.816
	0.490	267.037	1.086
	0.980	308.593	1.255
	1.960	392.277	1.595
	2.940	395.309	1.608
Y1	0.245	182.451	0.742
	0.490	213.433	0.868
	0.980	272.529	1.108
	1.960	300.888	1.224
	2.940	304.167	1.237
Y2	0.245	131.306	0.534
	0.490	182.041	0.741
	0.980	220.81	0.898
	1.960	258.431	1.051
	2.940	267.037	1.086
Y3	0.245	94.6678	0.385
	0.490	127.699	0.519
	0.980	167.615	0.682
	1.960	180.402	0.734
	2.940	196.057	0.797
Y4	0.245	70.898	0.288
	0.490	84.586	0.344
	0.980	140.404	0.571
	1.960	167.206	0.680
	2.940	183.762	0.747

One can see from Table 5 that K_{IC} increase with increasing loads meanwhile decreases with increasing gold addition. These behaviors can be seen more clearly in Fig. 1. A change in K_{IC} is related to a similar linear change in the average γ. This behaviors reason is like the one of E and Y. The B_i shows similar change with K_{IC}, increases with increasing load and decreases with increasing gold addition.

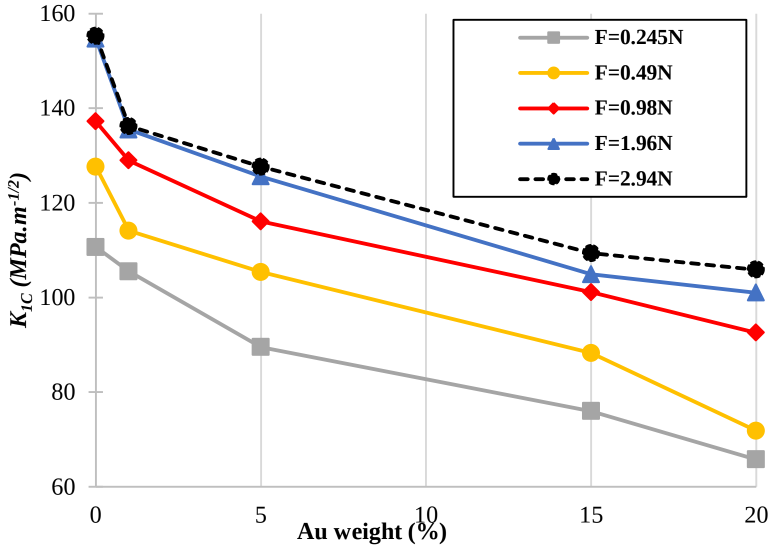

Figure 1. *K_{IC}–Au weight graph*

Table 5. *K_{IC} and B_i values of samples*

Sample	*F* *(N)*	*K_{IC}* *(kPa/$\sqrt{\mu m}$)*	*B_i* *($\sqrt{m}$)*
Y0	0.245	110.632	22.1274
	0.49	127.629	25.527
	0.98	137.201	27.4415
	1.96	154.689	30.9393
	2.94	155.287	31.0587
Y1	0.245	105.496	21.1002
	0.49	114.103	22.8216
	0.98	128.935	25.7882
	1.96	135.478	27.0967
	2.94	136.214	27.2439
Y2	0.245	89.497	17.9001
	0.49	105.378	21.0765
	0.98	116.058	23.2126
	1.96	125.556	25.1123
	2.94	127.629	25.527
Y3	0.245	75.992	15.199
	0.49	88.259	17.6526
	0.98	101.116	20.2242
	1.96	104.902	20.9814
	2.94	109.359	21.8729
Y4	0.245	65.763	13.1532
	0.49	71.832	14.3669
	0.98	92.545	18.5098
	1.96	100.993	20.199
	2.94	105.875	21.176

A change in E, Y, K_{IC} and B_i subtends to a change in the average γ. This result can also be obtained from the proposed hardness calculations. It is seen that the E, Y, K_{IC} and B_i of the samples in the present work indicate a strong dependency on applied load.

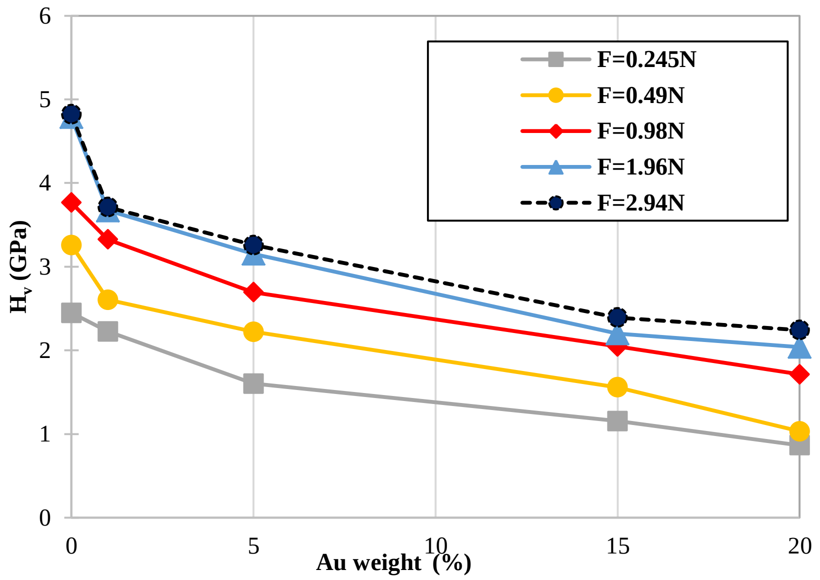

Figure 2. *H_v-Au weight graph for different F values*

In Fig. 2 we evaluate the effect of gold content and display the H_v as a function of the gold concentration at various applied loads. The H_v value decreases randomly as the percentage of gold increases from undoped to 20%. It is seen from Fig. 2 that adding gold causes a softening effect in the doped samples (Y1-Y4). The reason of degradation is explained by the formation of impurity phases and irregularities mainly distributed at the grain boundaries.

IV. CONCLUSION

This research presents the mechanical properties of gold added YBCO bulk superconductors. The samples were prepared by using the standard solid-state reaction method. Mechanical properties (E, Y, K_{IC} and B_i) of the samples have been reported for different load and gold concentration values. Gold added samples are collated with an undoped sample. The main findings of the study can be summarized as follows:

1- Gold addition improves the mechanical properties (flexibility) of YBCO superconductors which is an important parameter in superconductor cables.

2- The estimated E, Y, K_{IC} and B_i values of the samples depend on the changing applied load and gold addition. All four properties increase with an increase in the load.

3- Also, all four properties decrease with an increase in gold addition.

4- The load dependency of the E, Y, K_{IC} and B_i values, in contrast to ISE, exhibit a typical RISE.

5- It has been found that the calculated H_v is far from the H_0 value calculated using PSR model which means that the PSR model is not suitable for determination of the real microhardness value of gold added YBCO bulks [21].

V. REFERENCES

[1] S. Hahn *et al.*, "Design study on a 100-kA/20-K HTS cable for fusion magnets," *IEEE Trans. Appl. Supercond.*, vol. 25, no. 3, 2015.

[2] G. Celentano *et al.*, "Design of an industrially feasible twisted-stack HTS cable-in-conduit conductor for fusion application," *IEEE Trans. Appl. Supercond.*, vol. 24, no. 3, 2014.

[3] W.H. Fietz, R. Heller, S.I. Schlachter and W. Goldacker, "Application of high temperature superconductors for fusion," *Fusion Engineering and Design*, vol. 86, no. 6–8, pp. 1365-1368, 2011.

[4] W. Goldacker, F. Grilli, E. Pardo, A. Kario, S. Schlachter and M. Vojenčiak, "Roebel cables from REBCO coated conductors: a one-century-old concept for the superconductivity of the future," *Supercond. Sci. Technol.*, vol. 27, no. 9, 2014.

[5] S. B. Mohamed, S. A. Halim and H. Azhan, "Effect of doping Ba, Y, V, Zn and Sn on BSCCO superconducting ceramics," *IEEE Transactions on Applied Superconductivity*, vol. 11, no. 1, 2001.

[6] B. P. Mikhailov, G. S. Burkhanov, P. E. Kazin, V. V. Lennikov, S. V. Shavkin, G. V. Laskova and A. A. Titov, "Microstructure and superconducting properties of TaC-doped Bi-2223 ceramics," *Inorganic Materials*, vol. 37, no. 11, pp. 1199-1205, 2001.

[7] G. Yildirim, "Beginning point of metal to insulator transition for Bi-2223 superconducting matrix doped with Eu nanoparticles," *Journal of Alloys and Compounds*, vol. 578, pp. 526-535, 2013.

[8] M. Dogruer, Y. Zalaoglu, A. Varilci, C. Terzioglu, G. Yildirim and O. Ozturk, "A study on magnetoresistivity, activation energy, irreversibility and upper critical field of slightly Mn added Bi-2223 superconductor seramics," *J. Supercond. Nov. Magn*., vol. 25, pp. 961-968, 2012.

[9] G. Yildirim, S. Bal, E. Yucel, M. Dogruer, M. Akdogan, A. Varilci and C. Terzioglu, "Effect of Mn addition on structural and superconducting properties of (Bi, Pb)-2223 superconducting ceramics," *J. Supercond. Nov. Magn*., vol. 25, pp. 381-390, 2012.

[10] N. Soylu, C.C. Yahsi, S.P. Altintas, S. Nezir and C. Terzioglu, "The structural and electrical study of Lu-doped YBCO system," *J. Supercond. Nov. Magn.*, vol. 25, pp. 1945-1949, 2013.

[11] M.B. Turkoz, S. Nezir, C. Terzioglu, A. Varilci and G. Yildirim, "Investigation of Lu effect on YBa2Cu3O7-delta superconducting compounds," *Journal of Materials Science-Materials in Electronics*, vol. 24, no. 3, pp. 896-905, 2013.

[12] H. Sozeri, N. Ghazanfari, H. Ozkan and et al., "Enhancement in the high-T_c phase of BSCCO superconductors by Nb addition," *Supercond. Sci. Technol.*, vol.20, no. 6, pp. 522-528, 2007.

[13] M. M. A. Sekkina, H. A. El-Daly and K. M. Elsabawy, "Terbium-neodymium co-doping in Bi sites on the BPSCCO bismuth cuprate superconductor," *Supercond. Sci. Technol.*, vol. 17, no. 1, pp. 93-97, 2004.

[14] M. Dogruer, G. Yildirim, A. Varilci and C. Terzioglu, "MgB_2 inclusions in Bi-2223 matrix: the evaluation of microstructural, mechanical and superconducting properties of new system, Bi-2223 + MgB_2," *Journal of Alloys and Compounds*, vol. 556, pp. 143-152, 2013.

[15] R. Terzioglu, G. Aydin, N. Soylu Koc and C. Terzioglu, "Investigation of the structural, magnetic and electrical properties of the Au doped YBCO superconductors," *Journal of Materials Science: Materials in Electronics*, vol. 30, no. 3, pp. 2265-2277.

[16] H. Aydin, O. Cakiroglu, M. Nursoy, and C. Terzioglu, "Mechanical and superconducting properties of the $Bi_{1.8}Pb_{0.35}Sr_{1.9}Ca_{2.1}Cu_3Gd_xO_y$ system," *Chinese Journal of Physics*, vol. 47, no. 2, 2009.

[17] O. Ozturk, C. Terzioglu and I. Belenli, "Influence of diffusion-annealing temperature on the physico-mechanical properties of Au-doped Bi-2223 superconductors," *J Supercond Nov Magn*, vol. 24, pp. 381–390, 2011.

[18] O. Ozturk, E. Asikuzun, M. Erdem, G. Yildirim, O. Yildiz and C. Terzioglu, "The effect of Pr addition on superconducting and mechanical properties of Bi-2212 superconductors," *J Mater Sci: Mater Electron*, vol. 23, pp. 511–519, 2012.

[19] M. Yilmazlar, O. Ozturk, H. Aydin, M. Akdogan and C. Terzioglu, "The effect of Sm-Ca substitution on mechanical properties of BSCCO superconductors," *Chinese Journal of Physics*, vol. 45, no. 2, 2007.

[20] Y. Yoshino, A. Iwabuchi, K. Noto, N. Sakai and M. Murakami, "Vickers hardness properties of YBCO bulk superconductor at cryogenic temperatures," *Physica C: Superconductivity*, vol. 357–360, Part 1, pp. 796-798, 2001.

[21] R. Terzioglu, S. Polat Altintas, A. Varilci and C. Terzioglu, "Modeling of microhardness in the Au doped YBCO bulk superconductors," *Journal of Superconductivity and Novel Magnetism*, vol. 32, no. 11, pp. 3377-3383, 2019.

[22] A. Leenders, M. Ullrich, and H. C. Freyhardt, "Influence of thermal cycling on the mechanical properties of VGF melt-textured YBCO," *Physica C: Superconductivity*, vol. 279, no. 173, 1997.

[23] C. Veerender, V.R. Dumke and M. Nagabhooshanam, "Hardness and elastic moduli of $Bi_{2-x}Pb_xCa_2Sr_2Cu_3O_y$ superconductors," *Phys. Status Solidi A*, vol. 144, no. 199, 1994.

[24] F.A. McClintock, A.S. Argon and A. Frank, *Mechanical behavior of materials*, Sydney, Addison-Wesley Press, 1966, pp. 455.

[25] D. Tabor, *The hardness of metals*, Oxford, Clarendon Press, 1951.

[26] R.W. Davidge, *Mechanical behavior of ceramics*, Cambridge, Cambridge University Press, 1979, pp. 31–50.

[27] K. Hirao and M. Tomozawa, "Microhardness of SiO_2 glass in various environments," *J. Am. Ceram. Soc.*, vol. 70, no. 497, 1987.

[28] F. Frohlich, P. Grau and W. Grellmann, "Performance and analysis of recording microhardness tests," *Phys. Status Solidi A*, vol. 42, 1977.

[29] K. Nihara, R. Morena and D.P.H. Hasselman, "Evaluation of K_{IC} of brittle solids by the indentation method with low crack-to-indent ratios," *J. Mater. Sci Letters.*, vol. 1, no. 13, 1982.