

3rd International Conference on Innovative Academic Studies

September 26-28, 2023 : Konya, Turkey



All Sciences Proceedings <u>http://as-proceeding.com/</u>

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Decrement trend in mechanical performances and characteristic behavior of calcium saturated Bi-2212 Ceramics

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Abstract – This study focuses on the precise changes in basic mechanical design performance parameters (Vickers microindentation hardness, elastic modulus mechanical yield strength, fracture toughness, brittleness index, ductility, and elastic hardness coefficients) and mechanical characteristic features (typical indentation size effect or unconventional reverse indentation size effect). The investigation is carried out on Bi_{2.1}Sr_{2.0}Ca_{1.1}Cu_{2.0}O_v (Bi-2212) ceramic compounds with varying calcium impurity additions in the range of $0 \le x \le 0.1$. The materials are prepared through standard solid-state reaction methods. Experimental measurements are conducted using Vickers microindentation hardness testing with load intervals ranging from 0.245 N to 2.940 N. The results recorded show that the calcium impurity adversely affects the basic mechanical design performance parameters due to the increase in the permanent crystal defects and unrecoverable stress regions. Namely, the calcium ions lead to the reduction of the resistance against crack propagation, mechanical strength/durability, hardness, critic stress points, hardness, and resistivity towards to the applied force. Therefore, the increase in the calcium addition amount promotes rapid propagation of cracks and related defects, reaching terminal velocity for critical crack size at relatively lower test loads. As a result, the pure ceramic exhibits the highest mechanical performance features due to its least response and sensitivity to the applied test load while the Bi-2212 material produced with the maximum calcium oxide impurity level demonstrates the lowest performance characteristics under applied test forces. Additionally, all the ceramic materials produced exhibit standard indentation size effect (ISE) behavior due to the predominant character of recovery mechanisms over the compounds. However, the increase in Ca impurity amount disrupts the typical ISE property of the Bi-2212 ceramic system.

Keywords – Bi-2212 Ceramics; Cao Addition; Vickers Hardness Tests; Mechanical Performance; ISE.

I. INTRODUCTION

Resistance of a ceramic material to permanent irreversible microscopic structural shape changes is known as a measure of its resistance under applied test forces, commonly referred to as a microhardness parameter [1, 2]. In microhardness measurement tests, an indentation is heavily strained by using a controlled load to form an indentation on the specimen's surface. The notches formed within a cross-sectional geometry are numerically recorded, and the average notch values of the indentation dimensions are used to find the numerical values for the Vickers microindentation hardness parameters. Therefore, based on the penetration depth parameters of the indentation (to be used to convert into numerical values of Vickers microhardness parameters), it can be decided whether the material is soft or hard [3-6]. According to the determined Vickers microindentation hardness parameters, the indentation can penetrate much deeper and larger into the products examined. Conversely, provided that the material surface is sufficiently hard, it may leave shallow tracks on the indented specimen surface [7]. Thus, the question of "what do these measurement results serve for?" immediately arises.

Penetration depth is directly related to the degree of deformation, which is an essential parameter for determining the average economic service life and can be determined entirely through experimental mechanical measurement tests [8-11]. Specially, the determination of mechanical performance features of a hard (ceramic) material is very difficult as compared to the other relatively soft materials. In this respect. the variation in mechanical performance can also be examined through hardness determination tests conducted with the combination of very little force and a microscope. There are four main hardness tests: Brinell, Rockwell, Knoop, and Vickers hardness techniques [12, 13]. The latter method exhibits superior characteristics due to its non-destructive nature, straightforward test procedure, relatively low cost, damage-free results, quality control, accurate (reliable and useful) readings, and, especially, its independence from the test load applied to the measured hardness test [14].

In other words, Vickers microhardness results to find fundamental allow us mechanical performance quantities (microhardness, mechanical resilience, elastic modulus, mechanical yield strength, fracture toughness, shear modulus of rigidity, ductility, elastic hardness coefficient, shear modulus, flexibility, brittleness index, resilience, durability, critic stress points, and resistivity towards to the applied forces) and mechanical values, as well as characteristic features (typical indentation size effect or unconventional reverse indentation size effect). The findings are entirely beneficial parameters to decide on the economic service life and, particularly, application areas of a ceramic material [15, 16].

In the examination of this research, basic mechanical design properties and mechanical characteristic features of Bi-2212 ceramic compounds with different mole-mole ratios intervals $0.0 \le x \le 0.1$ of calcium impurity addition are differentiated through Vickers tests conducted at 0.245 N-2.940 N. Measurement evidence obtained demonstrates that increase in the level of new calcium impurity sites in bulk Bi-2212 ceramic material significantly harms general mechanical performance properties [8, 9].

II. MATERIALS AND METHOD

In the present work, the role of the calcium ion addition in the Bi-2212 superconducting system on the mechanical performance behavior of the $Bi_{2.1}Sr_{2.0}Ca_{1.1}Cu_{2.0}O_{v}$ ceramic material is investigated with precision using Vickers microhardness measurements. The effects of calcium atoms on mechanical performance and characterization are also thoroughly examined. Regarding the production process, high-purity chemicals (Bi₂O₃, SrCO₃, CaO, CaCO₃, and CuO) to prepare the Bi-2212 ceramic material are obtained from a specialized company in Ankara, Turkey. High-purity chemicals (\geq 99%) are weighed with precision to form a stoichiometric material with the $Bi_{2,1}Sr_{2,0}Ca_{1,1}Cu_{2,0}O_vCa_x$ composition. Subsequently, the powder of chemicals is mixed in an electronic grinder for approximately 6 hours to obtain a homogeneous mixture. The powder chemicals in homogeneous mixture are further ground in a mortar for about 30 minutes to have smaller and smaller particles and thus stronger bonds are formed between the foreign and host atoms. The homogeneous mixture with the optimal particle size is subjected to heat treatment in a muffle furnace at 800°C for duration of 24 hours. The heating and cooling rate during heat treatment is set at 5°C/min to avoid damage to the ceramic structure. Then, the homogenously calcined powders are removed from the furnace. Right after the last powder are pressed into rectangular prism shapes (1.5x0.5x0.2 cm³) and subjected to main heat treatment again at 840°C for 24 hours. Throughout this study, the Ca added Bi-2212 ceramic compounds produced and doped with different molar ratios (x=0.00, 0.01, 0.03, 0.05, 0.07, and 0.10) will be referred to as pure, Ca-1, Ca-2, Ca-3, Ca-4, and Ca-5.

The basic design mechanical performance and related characteristic (ISE or RISE beavior) property of pure and Ca-added Bi-2212 ceramics are properly examined by using Vickers microindentation hardness measurements performed under normal atmospheric pressure conditions. The different applied microindentation test loads varying from 0.245 N to 2.940 N are directly applied to the sample surfaces within 5 different locations for the duration of 10 seconds. Microhardness values for the samples were calculated using the equation $H_V = 1854.4(\frac{F}{d^2})$,

where F is the applied force and d is the diagonal length. After the measure of indentation diagonal lengths with the calibrated microscope, the microindentation hardness parameters including elastic (Young's) modulus (E), yield strength (Y), toughness (KIC), elastic fracture hardness coefficient (C11), brittleness index (B), and ductility (D) are determined [17]. Moreover, the microindentation hardness parameters are used to find the variation of mechanical characteristic features such as typical indentation size effect and untypical reverse indentation size effect with the calcium addition amount.

III. RESULTS AND DISCUSSION

The alteration in the fundamental mechanical design parameters of Bi-2212 ceramics due to calcium oxide impurity is experimentally analyzed through Vickers microindentation measurements performed at varied test forces beginning from 0.245 N until 2.94 N. The Vickers hardness measurement results are shown in Fig. 1. As observed that each ceramic compound exhibits different mechanical performances depending on both the level of calcium impurity addition and the test forces applied. Additionally, the increase in calcium impurity amount in the bulk Bi-2212 ceramic crystal structure leads to a significant change in microhardness parameters due to the emergence of new permanent crystal structure including microcrystal alignment issues. orientations, microvoids, cracks, and interaction problems between grains. In more detail, the Vickers hardness findings obtained from the current study in Fig. 1 indicate that the mechanical performance behavior of the Bi-2212 crystal system is reported to degrade systematically with increasing the calcium impurity amount and reach to the minimum constants for the Bi-2212 ceramic added by the highest calcium oxide powder (Ca-5 sample). Scientifically, the Ca ions damage the formation of slip systems, and thus the mobility of dislocations, defects, and cracks increases as much as possible. In other words, the calcium ions result in the decrement in the resistance against crack propagation, leading to a reduction of the mechanical strength/durability, hardness, critic stress points, hardness, and resistivity towards to the applied force. Accordingly, there appears a phase transition from durable tetragonal phase to stressaugmented phase in the excess calcium saturated Bi2212 products, triggering crack nucleation in permanent and unrecoverable stress regions, stress concentrators, concentration and zones. Consequently, it is noted that the fracture strength decreases as the Ca impurity amount increases in the system. Furthermore, the dramatic degradation is observed in the hardness data depending on the enhancement in the test forces applied. In this context, the applied test load also encourages the increase in stress concentrators and concentration zones in Bi-2212 superconductors. With the increase in stress concentration regions, dislocations, cracks, and related defects propagate rapidly and reach terminal velocity for critical crack size at a relatively lower test load based on the increase of granularity degree and internal strain energy of the system.



Moreover, the experimental findings provided in Fig. 1 clearly reveal that the pure and Ca added new Bi-2212 compounds possess the standard indentation size effect (ISE) behavior because of the non-linear decrease in the Vickers microindentation hardness parameters with the applied forces. Correspondingly, both the elastic/reversible and plastic/irreversible faults (deformations) appear instantaneously due to the predominant character of a recovery mechanism. Nevertheless, the hardness data in Fig. 1 show that the ISE nature tends to decrease with the enhancement in the calcium oxide addition amount in the Ca-site doped Bi-2212 system due to the increase in the new permanent crystal structure problems and stress concentration zones. In this respect, the Bi-2212 ceramic with the largest calcium oxide addition amount is noted to present the worst ISE behavior. Further, the experimental findings embedded in the figure reveal that the pure material provides the least response and sensitivity to the test loads applied. Oppositely, the calcium oxide ions encourage the response and sensitivity of Ca added new Bi-2212 crystal system.

With help of the variation in the microindentation hardness parameters against the test forces applied, we inspect the fundamental design performance features such as microhardness (H_v) , elastic modulus (abbreviated as E, representing resistance to plastic deformation), mechanical yield strength (starting point of permanent plastic deformation and referred to as Y), fracture toughness (shown as K_{IC}, the critical stress intensity factor at which the propagation of a crack becomes suddenly rapid and unlimited, characterized by a sharp crack), brittleness index (presented as B; related to the ratio between the tensile and compressive strengths), ductility (a mechanical property typically defined as a material's susceptibility to being stretched and defined as the ability of a material to withstand plastic deformation under tensile stress before breaking), and elastic hardness coefficient (C_{11}) through actual microindentation hardness values using the following relationships (1-6):

$$E = 81.9635H_V$$
(1)

$$Y \approx \frac{n_V}{3} \tag{2}$$

$$K_{IC} = \sqrt{2E\alpha} (a \text{ related to surface energy parameter})$$
(3)
$$B = \frac{H_v}{(4)}$$

$$D = \frac{1}{2}$$
(5)

$$C_{11} = H_{\nu}^{\frac{7}{4}}$$
(6)

$$\mathbf{C}_{11} = \mathbf{H}_{v}$$

Table 1 presents all the calculated real indentation hardness evidence. Based on mechanical results provided in the table, it is noted that all fundamental design parameters undergo significant changes with increasing levels of calcium oxide impurity amount and test forces applied. Roughly, it can be seen from the table that all properties (except for the ductility) such as microhardness, elastic modulus, mechanical yield strength, elastic hardness coefficient, and brittleness index parameters are observed systematically decrease with increasing levels of calcium oxide impurity amount. The similar situation is seen form the increase in the test forces applied. The decrease in the non-linear reduction with the applied test load stems from the general mechanical characteristic of ISE behavior. To say that the Vickers hardness data decrease non-linearly with an increase in the test load magnitude [18-20] (even having seen in Fig. 1).

Table 1 Variation of H_{ν} , E, Y, U_r, and G parameters as a function of applied test load

Samples	F (N)	d (µm)	H _v (GPa)	E (GPa)	Y (GPa)
Pure	0.245	30.62041	0.48456	39.71623	0.16152
	0.49	44.13075	0.46657	38.24171	0.15552
	0.98	63.18842	0.45515	37.30569	0.15172
	1.96	89.73035	0.45142	36.99996	0.15047
	2.94	110.19745	0.44896	36.79833	0.14965
	0.245	31.28674	0.46414	38.04254	0.15471
Ca-1	0.49	45.22629	0.44424	36.41147	0.14808
	0.98	64.94966	0.4308	35.30988	0.14360
	1.96	92.34163	0.42625	34.93694	0.14208
	2.94	113.34250	0.42439	34.78449	0.14146
Ca-2	0.245	31.89378	0.44664	36.60818	0.14888
	0.49	47.31639	0.40825	33.26571	0.13529
	0.98	68.10295	0.38706	32.11576	0.13061
	1.96	96.92629	0.38389	31.71004	0.12896
	2.94	118.90839	0.38623	31.60431	0.12853
Ca-3	0.245	32.88776	0.42005	34.42877	0.14002
	0.49	48.40625	0.38779	31.78463	0.12926
	0.98	69.90018	0.37194	30.48550	0.12398
	1.96	99.57930	0.36654	30.04290	0.12218
	2.94	122.03250	0.3661	30.00684	0.12203
	0.245	32.98133	0.41767	34.23370	0.13922
Ca-4	0.49	48.99540	0.37852	31.02482	0.12617
	0.98	71.17258	0.35876	29.40523	0.11959
	1.96	101.54161	0.35251	28.82328	0.11722
	2.94	124.51277	0.35166	28.89295	0.11750
Ca-5	0.245	34.02240	0.3925	32.17067	0.13083
	0.49	51.33605	0.34479	28.26020	0.11493
	0.98	75.07762	0.32241	26.42585	0.10747
	1.96	107.32711	0.31553	25.86194	0.10518
	2.94	131.58810	0.31486	25.80703	0.10495

	Table 1 continues.							
Samples	F (N)	Кіс (MPam ¹ ^{/2})	<i>C</i> ₁₁ (<i>GPa</i>) ^{7/4}	B (m ^{1-1/2})	$D(m^{1/2})$			
Pure	0.245	0.24294	0.28142	1.99457	0.50136			
	0.49	0.23838	0.26339	1.95725	0.51092			
	0.98	0.23545	0.25221	1.93311	0.51730			
	1.96	0.23448	0.24861	1.92520	0.51943			
	2.94	0.23384	0.24624	1.91995	0.52085			
Ca-1	0.245	0.25446	0.26100	1.82402	0.54824			
	0.49	0.24894	0.24173	1.78453	0.56037			
	0.98	0.24515	0.22908	1.75729	0.56906			
	1.96	0.24385	0.22486	1.74800	0.57208			
	2.94	0.24332	0.22315	1.74416	0.57334			
	0.245	0.29268	0.24402	1.52604	0.65529			
Ca-2	0.49	0.27900	0.20638	1.45470	0.68743			
	0.98	0.27414	0.19405	1.42931	0.69964			
	1.96	0.27240	0.18978	1.42026	0.70410			
	2.94	0.27195	0.18868	1.41787	0.70528			
	0.245	0.27894	0.21917	1.50588	0.66406			
Ca-3	0.49	0.26802	0.19057	1.44687	0.69115			
	0.98	0.26248	0.17714	1.41702	0.70571			
	1.96	0.26057	0.17267	1.40669	0.71089			
	2.94	0.26041	0.17231	1.40586	0.71131			
Ca-4	0.245	0.30738	0.21700	1.35881	0.73594			
	0.49	0.29262	0.18267	1.29355	0.77307			
	0.98	0.28488	0.16631	1.25934	0.79407			
	1.96	0.28205	0.16059	1.24680	0.80205			
	2.94	0.28239	0.16127	1.24831	0.80108			
Ca-5	0.245	0.32484	0.19463	1.20829	0.82762			
	0.49	0.30446	0.15514	1.13246	0.88303			
	0.98	0.29441	0.13795	1.09511	0.91315			
	1.96	0.29125	0.13284	1.08336	0.92305			
	2.94	0.29094	0.13234	1.08222	0.92403			

This is because, the dominant character for recovery (dependent on surface energy) is prevalent for all Bi-2212 superconducting materials, although it shows a consistent decreasing trend with calcium oxide impurity addition [21-23].

When evaluating all experimental findings individually, the un-added Bi-2212 sample exhibits the highest microhardness parameters at any applied test load. Numerically, for the pure sample, the maximum H_v value of approximately 0.48456 GPa is observed at the lowest applied test force (0.245)N), while the parameter is found to decrease from 0.48456 GPa to 0.44896 GPa at the highest indentation test force of 2.940 N. In the case of the maximum calcium oxide addition level, the Ca-5 sample presents the smallest values, ranging from 0.39250 GPa to 0.31486 GPa (Table 1). The initial value is observed at the lowest applied test load of 0.245 N, and the subsequent value is obtained at the highest indentation test load of 2.940 N. Consequently, the results indicate that the pure material possess the smallest responsibility and sensitivity to forces applied. Conversely, the bulk Ca-5 ceramic sample is the most responsive to the test forces applied.

As for the Young's modulus features, it is found from Table 1 that the un-doped Bi-2212 product is observed to have the elastic modulus parameters under any applied test forces. In this respect, the E value is obtained to be range from 39.71623 GPa (at the force of 0.245 N) to 36.79833 GPa (at the 2.940 N test force) by using Eq. 1. In the case of the highest addition amount, the Ca-5 sample has the lowest E values between 32.17067 GPa (under 0.245 N) to 25.80703 GPa (at 2.940 N). The other ones are noticed to possess the moderate parameters. All in all, the pure sample (prepared without excess calcium oxide chemicals) exhibits the highest resistance to plastic deformation.

Thirdly, we investigate the variation in mechanical yield strength parameters with calcium oxide addition amount and applied test forces in detail based on the experimental results extracted from Eq. 2. According to the yield strength parameters given in Table 1, the pure sample is realized to obtain the maximum Y values at any applied test load, while the minimum mechanical yield strength parameters are recorded for the Ca-5 ceramic compound under 2.940 N applied force. Numerically, it is observed that the maximum parameter is approximately 0.16152 GPa for the pristine ceramic under 0.245 N. In contrast, the Ca-5 sample obtains a minimum Y value of 0.13083 GPa at the same applied load. When the maximum microindentation test load is 2.940 N, the previous material exhibits a Y value of 0.14965 GPa, while the global minimum of 0.10495 GPa is attributed to the latter Ca-5 ceramic material. Accordingly, the un-substituted ceramic has maximum starting point of permanent plastic deformation. Therefore, the findings for the elastic modulus and yield strength parameters are observed to support each other.

Fourthly, we examine the role of extra Ca-sites in the Bi-2212 superconducting ceramic structre on the fracture toughness (K_{IC}) parameters or critical stress intensity factor by means of the Vickers hardness findings using Eq. 4. One can encounter every K_{IC} constant of pure and Ca-side added Bi-2212 system in Table 1. As seen that K_{IC} calculations are noted to decrease with increase in the forces applied. In this respect, the pure sample has 0.24294 MPam^{1/2} under the minimum force of 0.245 N. Oppositely, the parameter is found to decrease regularly and fall in its minimum value of 0.23384 MPam^{1/2} at the maximum microindentation test load of 2.940 N. Interestingly, the extra calcium addition leads to increase in the K_{IC} parameters. Accordingly, the Ca-5 has the maximum K_{IC} constant of 0.32484

 $MPam^{1/2}$ at 0.245 N applied force. Similar to the pure sample findings, with the increase in the applied test force, the parameter is recorded to reduce to the value of 0.29094 $MPam^{1/2}$.

At the same time, defining the variation in the brittleness index (B) and ductility (D) features is of important to discuss the effect of calcium oxide addition in the Bi-2212 ceramics. All the calculations for the B and D parameters are embedded in Table 1. It is obvious that the parameters behave exactly in reverse. In more detail, like the H_v, E, and Y findings, the pure ceramic has the largest B coefficient of 1.99457 m⁻ $^{1/2}$ at 0.245 N applied force whereas the value is found to decrease towards 1.91995 m^{-1/2} at 2.940 N applied force because of the ISE characteristic nature. With ascending calcium oxide in the Bi-2212 material, the B parameter tends to decrease rapidly. On this basis, the Ca-5 obtains the smallest values at any applied forces. In the case of 0.245 N, the parameter is noted to be about 1.20829 m^{-1/2}. On the other hand, under the maximum applied test load, B decreases to the 1.0822 m^{-1/2}. As explained above, the ductility moves totally in reverse. Thus, the maximum ductility value of $0.92403 \text{ m}^{1/2}$ for the Ca-5 sample under 2.940 N applied force. Conversely, the smallest value of $0.50136 \text{ m}^{1/2}$ for the un-substituted product at 0.245 N applied force.

Lastly, we focus strongly on the influence of calcium oxide addition amount on the elastic hardness coefficient (C₁₁) parameters for the Caadded Bi-2212 crystal systems. Based on Eq. 6, we calculate the C₁₁ coefficients and provided in Table 1 in detail. As encountered, the C_{11} coefficients are observed to diminish monotonously with the increase in both the applied test forces and calcium oxide impurity in the system. Thus, the maximum the values of C_{11} coefficients is obtained to be about 0.28142 (GPa)^{7/4} for the un-substituted sample at an applied force of 0.245 N. However, the pure sample exhibits the minimum value of 0.24624 (GPa)^{7/4} under 2.940 N applied force. As for the role of calcium addition amount on the C_{11} coefficients, the Ca-5 sample is noted to have the smallest C₁₁ values between 0.19463 (GPa)^{7/4} and 0.13234 (GPa)^{7/4}.

The diminish of mechanical performance properties depending on the increase in the Ca impurity addition and applied test force stems from the reduction of recovering elastic energy capability after unloading. Thus, it can be put forward that the pure/Ca-5 ceramic compound provides the least/most response and sensitive to the applied test loads. All in all, the experimental results show that the reduction of key mechanical performance parameters as against the Ca impurity addition amount is due to the new induction of crystal structure problems including microcrystal coalescence orientations, microvoids, cracks, and interaction problems between grains [24, 25]. Besides, the degradation in the parameters with the applied test loads is the result of a typical property of the ISE feature.

IV. CONCLUSION

In this study, we investigate the differentiations in the basic performances and overall mechanical characteristics of the Bi-2212 ceramic structures with varying levels of new Ca-sites using Vickers microhardness experimental measurements applied at load ranges of 0.245 N to the value of 2.940 N. Experimental measurements allow us to determine essential mechanical design parameters such as Vickers microindentation hardness, elastic modulus mechanical yield strength, fracture toughness, brittleness index, ductility, and elastic hardness coefficients in detail for the first time. Furthermore, we discuss mechanical characteristic features such as the typical indentation size effect and reverse indentation size effect, which are dependent on the surface energy-based elastic recovery mechanism. It is observed that all significant mechanical design parameters are noted to suppress as the calcium oxide impurity increases in the system because of ascending the permanent crystal structure faults and reduction of slip systems along with crystal structure. Hence, the calcium ions cause to the diminish in the resistance against crack propagation, mechanical strength/durability, hardness, critic stress points, hardness, and resistivity towards to the applied force. Besides, the Ca ions lead to the phase transition from durable tetragonal phase to stressinduced phase in the excess calcium saturated Bi-2212 products. To sum up, while the pure sample exhibits the highest performance findings of H_v, E, Y K_{IC}, B, and C₁₁ properties against the forces applied, the Bi-2212 ceramic compound prepared with the maximum Ca addition amount offers the lowest performance characteristics. Additionally, both the pure and Ca added Bi-2212 ceramics exhibit a standard ISE nature, but in decrement character as the addition amount increases. Similarly, the recovery mechanism is found to follow the same trend (decrease with the addition level and applied test forces). Further, the pure material presents the least response and sensitivity to the loads applied while the bulk Ca-5 product is the most sensitive and responsive to the applied microindentation test load.

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