All Sciences Proceedings
http://as-proceeding.com/

3rd International Conference on Innovative Academic Studies

September 26-28, 2023 : Konya, Turkey

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Dependence of General Mechanical Properties of Bi-2212 Ceramics on Calcium Oxide

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Abstract – In this study, we investigated the role of calcium oxide (CaO) impurity addition and applied micro-indentation test loads on some mechanical performance parameters including elastic moduli (E), shear (*G*) modulus and resilience (U_r) of $Bi_{2.1}Sr_{2.0}Ca_{1.1}Cu_{2.0}O_y$ ceramics using Vickers hardness test measurements. The tests were conducted in test forces ranges from 0.245 N to 2.940 N. Additionally, experimental findings reveal the relationship between Vickers results and test forces applied, and specifically identify the impact of Ca ion impurity on the elastic modulus for Bi-2212 ceramics. All experimental findings indicated a strong dependence of the examined mechanical performance quantities on both the loads applied and the amount of calcium oxide impurity addition. As the level of calcium impurity addition increased in bulk Bi-2212 ceramic compounds, it was noted that mechanical performances are systematically deteriorated due to an increase in microvoids, strains, prevalent defects, bonding problems, and crystallinity faults based on interaction between adjacent layers of ceramics. Furthermore, increasing the applied test force damaged the mechanical performances of Bi-2212 ceramics due to their granular structure. Thus, it was indicated that the pure sample has the best crystal quality while the Bi-2212 ceramic prepared with the maximum CaO addition amount of x=0.1 has the maximum crystal structure problems. In this regard, the former ceramic exhibited much stronger mechanical performance quantities compared to the other prepared ceramics. In other words, the pure ceramic material showed the least response and sensitivity to the microindentation test load. In summary, the idea of adding calcium oxide impurity fails to improve the mechanical performance, durability of the tetragonal phase, and stabilization of the Bi-2212 superconducting system.

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

I. INTRODUCTION

The discovery of zero-resistance superconductivity phenomenon [1-3] has directly guided academic researchers in the field of superconducting materials towards fundamental technological, sensitive process control, industrial, medical, high-energy, electro-optical, power transmission cable applications, spintronics, medical diagnostics, renewable energy, levitated trains, motors, the future hydrogen society, particle accelerators, cooling, and large-scale application areas [4-9]. Studies, particularly on Type II superconducting ceramic compounds (following the discovery of ceramic superconducting material $La_{2-x}Ba_xCuO_4$ [10, 11]) have garnered significant attention in the materials research community. Among the Type II superconducting ceramic compounds, the Bi-based ceramic family, with its main three participants, possesses the capability of carrying larger critical current density, higher critical transition temperature (onset and offset), lower energy loss, minimal power losses, relatively larger critical current (≈10^7A/cm^2), and a higher magnetic field-carrying capacity (≥100 T at 35 K) [12-16]. Similarly, another feature of superconducting materials containing Bi is the absence of power consumption, allowing them to be used in a much wider range of applications.

As the fundamental characteristic properties, such as electrical, superconductivity, flux pinning, crystalline quality, microstructural, physical, modulation, pairing mechanism, crystallization reaction kinetics, nucleation stability, crystallization temperature, discrete observation-like formation, morphological, slip system, basic mechanical performance (microhardness, mechanical resilience, elastic modulus, mechanical yield strength, fracture toughness, shear modulus of rigidity, ductility, elastic hardness coefficient, brittleness index, shear modulus, flexibility, resilience, durability, critic stress points, and resistivity towards to the applied forces), and mechanical characteristic properties, continue to improve, these types of superconducting materials find even more prominent places in potential application areas.

In this study, we examined the effect of calcium oxide impurity addition amount ($0 \leq x \leq 0.1$) on some important mechanical performance quantities (durability, resistivity towards to the applied forces, resilience, and shear moduli) using traditional Vickers microhardness tests performed at various applied microindentations with test loads ranging from 0.245 N to 2.940 N. Simultaneously, we determined the relationship between the mechanical performance behavior and the calcium impurity addition level as well as the applied test forces. Experimental results revealed clearly that that an increase in both applied test loads adversely affects the mechanical performance characteristics of Bi-2212 superconducting ceramic compounds due to an increase in microvoids, strains, prevalent defects, bonding problems, and crystallinity faults based on interaction between adjacent layers of ceramics.

II. MATERIALS AND METHOD

In this study, we investigated the influence of calcium oxide particles addition in the Bi-2212 superconducting system on the mechanical performance of the $Bi_{2.1}Sr_{2.0}Ca_{1.1}Cu_{2.0}O_y$ ceramic material depending on Vickers microhardness measurements. We also thoroughly examine the effects of extra Ca-sites on mechanical performance and characterization of Bi-2212 ceramics. For the production process, we purchased high-purity chemicals (CaO, $SrCO_3$, Bi_2O_3, $CaCO_3$, and CuO) to prepare the Ca added Bi-2212 new superconducting matrix. These high-purity chemicals (≥99%) are precisely weighed to form a stoichiometric ceramic material with the composition of $Bi_{2.1}Sr_{2.0}Ca_{1.1}Cu_{2.0}O_y+Ca_x$ system. The powdered chemicals were then mixed in an electronic grinder for approximately 6 hours to ensure a uniform mixture. After that, this mixture was further ground in a mortar for about 30 minutes to reduce particle size and strengthen the bonds between foreign and host atoms so that the ceramic phase can be formed easily. The resulting homogeneous mixture with optimal particle sizes underwent heat treatment in a furnace at the temperature of 800°C for time of 24 hours. During the calcination treatment, the heating and cooling rate were set at 5°C/min to prevent damage to the general ceramic structure of Bi-2212 system. After the heat treatment, the homogenously calcined powders were removed from the furnace for preparing the main heat treatments.

Subsequently, the calcined powders were pressed into rectangular prism shapes (15x5x2 mm^3) and subjected to main heat treatment at 840°C for 24 hours for sintering process. The Bi-2212 ceramics with varying molar ratios of Ca (x=0.00, 0.01, 0.03, 0.05, 0.07, and 0.10) were referred to as pure, Ca-1, Ca-2, Ca-3, Ca-4, and Ca-5 ceramics.

The mechanical performance properties as regards shear modulus and resilience parameters of pure and Ca-added Bi-2212 ceramics were thoroughly examined using Vickers microindentation hardness measurements conducted under normal atmospheric pressure conditions [17]. Varied microindentation test forces ranging from 0.245 N to 2.940 N were directly applied to the sample surfaces at five different locations for a duration of 10 seconds so that we could determine the average indentation value of notch track appeared on the surfaces. We also investigated the link between microhardness findings and test forces to the influence of calcium oxide on the formation of crystal structure problems, and the response, sensitive, and resistance to the applied forces.

III. RESULTS AND DISCUSSION

A. Mechanical Performances of Ca Added Bi-2212 System Based on Vickers Hardness Measurements

The crucial changes occurring in the fundamental mechanical features of Bi-2212 ceramics due to calcium oxide impurities within the varied molar addition ratio in a range of $0 \leq x \leq 0.1$ were experimentally analyzed through Vickers micro-indentation measurements conducted at test forces ranging from 0.245 N to 2.94 N. The Vickers hardness measurement results were graphical depicted in Fig. 1. As observed, each ceramic compound exhibits different mechanical performances depending on both the level of calcium impurity addition and the applied test forces. Furthermore, the increase in the amount of calcium oxide impurity in the bulk Bi-2212 ceramic crystal structure leads to a significant alteration in micro hardness parameters due to the emergence of new persistent crystal structure faults including microvoids, strains, prevalent defects, bonding problems, and crystallinity faults based on interaction between adjacent layers of ceramics. Thus, with the increase in the calcium oxide amount in the system the mechanical properties were obtained to suppress dramatically. Namely, the addition of calcium oxide into the Bi-2212 system fails to improve the general hardness parameters including shear (G) modulus and resilience (U_r) parameters. The effect of calcium oxide addition on the shear modulus (measurement of rigidity modulus and elastic shear hardness) and resilience (ability to absorb and release energy after reversible deformation) parameters based on the microindentation test results provided in Fig. 1 for the Bi-2212 ceramic system by using the following equations.

$$E = 2G(1 + \upsilon) \tag{1}$$

$$U_r = Y^2/2E \tag{2}$$

The inner constant of a material can be calculated from Poisson's ratio embedded in the strong formula including the elastic and shear moduli. According to Eq. 1 given above, the shear moduli are directly related to the modulus of elasticity for the material studied, and the Poisson ratio (abbreviated as υ) is used to be 0.25 in the equation [18]. The value of the Poisson ratio stems from the random orientation of superconducting adjacent layers in a Bi-2212 system with an isotropic structure. The computed shear moduli for the pure and calcium oxide added Bi-2212 ceramics were numerically depicted in Table 1.

Table 1 Differentiation of shear modulus and resilience values against the test forces applied.

Samples	*F (N)*	*G(GPa)*	*U_r (MPa)*	*E (GPa)*
Pure	0.245	15,88649	0,32840	39,71623
	0.49	15,29668	0,31620	38,24171
	0.98	14,92228	0,30850	37,30569
	1.96	14,79998	0,30600	36,99996
	2.94	14,71933	0,30430	36,79833
Ca-1	0.245	15,21702	0,31460	38,04254
	0.49	14,56459	0,30110	36,41147
	0.98	14,12395	0,29200	35,30988
	1.96	13,97478	0,28890	34,93694
	2.94	13,91380	0,28760	34,78449
Ca-2	0.245	14,64327	0,30270	36,60818
	0.49	13,30628	0,27510	33,26571
	0.98	12,84630	0,26560	32,11576
	1.96	12,68402	0,26220	31,71004
	2.94	12,64172	0,26140	31,60431
Ca-3	0.245	13,77151	0,28470	34,42877
	0.49	12,71385	0,26280	31,78463
	0.98	12,19420	0,25210	30,48550
	1.96	12,01716	0,24840	30,04290
	2.94	12,00274	0,24810	30,00684
Ca-4	0.245	13,69348	0,28310	34,23370
	0.49	12,40993	0,25660	31,02482
	0.98	11,76209	0,24320	29,40523
	1.96	11,52931	0,23840	28,82328
	2.94	11,55718	0,23890	28,89295
Ca-5	0.245	12,86827	0,26600	32,17067
	0.49	11,30408	0,23370	28,26020
	0.98	10,57034	0,21850	26,42585
	1.96	10,34478	0,21390	25,86194
	2.94	10,32281	0,21340	25,80703

It is visible that the G parameters are noted to decrease regularly with increasing the CaO impurity amount. This is because, the calcium ions strongly harmed the rigidity modulus and elastic shear characteristics of Bi-2212 system. It is another probable result deduced that increasing the applied force on the specimen surface led to the degradation in the G parameters due to the complete indentation size effect (ISE) character. Numerically the variation of G coefficients with the calcium oxide addition amount was found to be much stronger than that of applied test forces. In this respect, the maximum shear modulus value of

15.88649 GPa belonged to under the minimum test force of 0.245 N for the un-added ceramic. With the increase in the indentation test load, the G parameter got smaller and smaller, in fact, the coefficient reached the smallest value of about 14.71933 GPa for the pure material. Among all the values obtained, the minimum value of 10.32281 GPa was found for the Ca-5 ceramic sample at the 2.940 N applied test force. The decrease in the G moduli parameters resulted from the increase in granularity degree (induction of crystallinity problems), and orientation and geometry of cracks. The other ceramics were found to present the moderate coefficients between 15.21702 GPa at 0.245 N applied force for the Ca-1 compound and 11.55718 under 2.940 N test force for the Ca-4 material.

Additioanlly, the influence of calcium oxide impurity addition on the resilience parameters was associated with the capability of recovering elastic energy after unloading. In other words, resilience was directly related to the absorb energy capacity of a material. The elastic region areas under the stress-strain plots pointed out elastically absorbed energy per unit volume of ceramics and can be computed from Eq. 2 provided above. The left-hand side of the relation showed the resilience modulus while on the right-hand side Y abbreviated the yield strength, and E revealed the modulus of elasticity of every ceramic compound. One can see all the calculation values in Table 1. It is obvious that as the Ca oxide impurity amount increases in the Bi-2212 ceramics the U_r constants were noted to decrease systematically at any applied test load. On this basis, the pure ceramic possessed the maximum U_r parameter of 0.32840 MPa at the minimum applied force of 0.245 N followed by a systematic reduction of the values towards to 0.30430 MPa under the 2.940 N applied test force. As for the U_r data of other materials (Ca-1, Ca-2, Ca-3, and Ca-4 ceramics), among the compounds, the Ca-5 sample with the extra Ca-sites exhibited the smallest value of 0.21340 MPa under a 2.940 N applied test force. The decrease in the resilience parameters depending on the increase in the calcium oxide impurity addition and applied test force stemmed from the reduction of recovering elastic energy capability after unloading. Thus, it can be put forward that the pure/Ca-5 ceramics provided the least/most response and sensitive to the applied test forces. All in all, the experimental results showed that the reduction of mechanical performances as against the calcium oxide impurity addition amount was due to the increase in microvoids, strains, prevalent defects, bonding problems, and crystallinity faults based on interaction between adjacent layers of ceramics. Besides, the degradation in the parameters with the applied test loads is the result of a typical property of the ISE feature.

B. Connection between Vickers Hardness Findings and Test Forces

As per the mechanical performance findings given in Section 1 of this article, it pointed out that there was a strong correlation between the applied test loads and the Vickers microindentation hardness parameters due to variations in the main mechanical performance quantities caused by the addition of calcium oxide particles. This strong connection can be demonstrated by fitting equations from three-degree formulas to reach the maximum correlation values of R^2=0.9872 and 0.9691 as depicted in Fig. 1. All the fitting relationships were provided in Table 2.

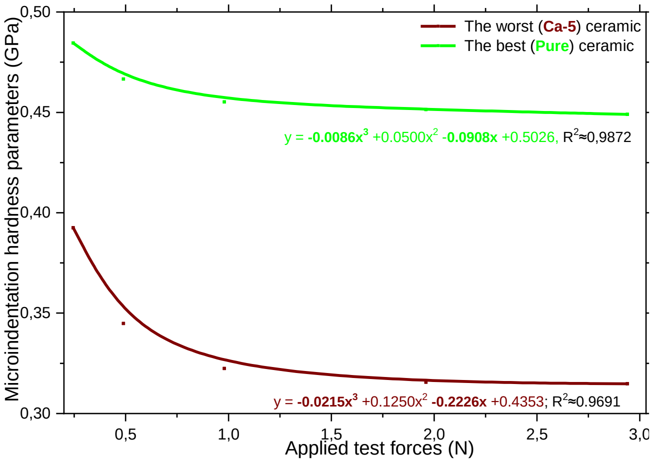

Figure 1 Sensitivity to the test forces applied for ca added Bi-2212 ceramics

From the table, variables were found to be negative and positive. The negative findings resulted from the typical ISE characteristic nature. Moreover, it was evident that all terms, including x^3, x^2, and x, are significantly dependent on the amount of calcium oxide impurity added. The variation of independent variables indicated the successful incorporation of calcium impurity into the $Bi_{2.1}Sr_{2.0}Ca_{1.1}Cu_{2.0}O_y$ ceramics crystal lattice. Furthermore, it was noticed that the dependency of the parameter x^3, and x^1 (indicator of ISE characteristic bahavior) systematically increased from -0.0086x^3 for the pure sample to -0.0215x^3.

Table 2. Connection between hardness properties and test forces applied for all Ca added Bi-2212 ceramics

Samples	*Fitting equations for Ca added Bi-2212 ceramic samples
Pure	$y = -0.0086x^3 + 0.0500x^2 - 0.0908x + 0.5026$
Ca-1	$y = -0.0094x^3 + 0.0550x^2 - 0.1014x + 0.4844$
Ca-2	$y = -0.0179x^3 + 0.1027x^2 - 0.1791x + 0.4800$
Ca-3	$y = -0.0144x^3 + 0.0840x^2 - 0.15118x + 0.4492$
Ca-4	$y = -0.0177x^3 + 0.1033x^2 - 0.1854x + 0.4536$
Ca-5	$y = -0.0215x^3 + 0.1250x^2 - 0.2226x + 0.4353$

The increase in the x^3 parameter was attributed to the escalation of problems in the crystal structure such as microvoids, strains, prevalent defects, stresses, bonding problems, and crystallinity faults based on interaction between adjacent layers of ceramics. Consequently, it was clear that the pure sample with relatively stronger mechanical performance quantities exhibited the least response and sensitivity to the applied test force while the Bi-2212 prepared with the maximum calcium impurity addition amount showed the highest response and sensitivity to the test forces applied due to its inferior crystal quality.

C. Role of Calcium Oxide Impurity in Elastic Modulus for Bi-2212 Ceramic System

In this section, we focused on the variation of the porosity degree (porosity) concerning the elastic modulus of calcium oxide added Bi-2212 samples prepared under applied test forces for determining the role of applied test loads on the porosity degree that is responsible for potential engineering and industrial applications of ceramic compounds [18]. On this basis, we investigated the role of calcium oxide particles addition and applied test forces on the elastic modulus of the bulk Bi-2212 ceramic structure using the granularity degree (porosity) deduced from the Vickers microindentation hardness tests [19]. The relationship between the Young's modulus and porosity is utilized to determine the relative degrees of porosity for pure and Ca added Bi-2212 ceramics as given below [19]:

$$E = E_0(1 - 1.9P + 0.9P^2) \quad (3)$$

According to the Vickers hardness findings given in Table 1, the parameter E_0 for the pure sample is determined to be approximately 39,71623 GPa. The highest elastic modulus parameter (indicating the densest material) was obtained from measurements conducted with a 0.245 N microindentation test force. Other elasticity moduli can also be seen in Table 1. The relative degrees of porosity parameters calculated based on the Young's modulus parameter are provided numerically in Table 3.

According to the table given below, it is evident that the relative degrees of porosity parameters were significantly dependent on both the level of calcium impurity addition amount and the test forces applied. More specifically, it was found that the degree of porosity was noted to decrease significantly with an increase in the level of calcium impurity addition amount. In this regard, it was noted that the minimum relative porosity value was around 1.7866% for the Ca-1 sample under applied 0.490 N microindentation test force. This parameter was calculated to systematically increase with the calcium oxide addition amount. For the bulk Ca-5 ceramic sample, a relative porosity value of approximately 18.4463% was observed under the same test load. Similarly, for the Ca-5 material under the applied 0.245 N microindentation test load, it had a porosity value of 13.6252%. Furthermore, an increase in the applied test load led to an increase in the degree of porosity. Based on this, it is observed that the maximum porosity parameter for the Ca-5 sample with the most granular structure under the applied 2.940 N applied force was recorded to be approximately 22.6452%.

The findings indicated that both the level of calcium impurity addition amount and the applied test force triggered the induction of microvoids, strains, prevalent defects, stresses, bonding problems, and crystallinity faults based on interaction between adjacent layers of ceramics [20]. Consequently, mechanical performances and the stabilization of the durable tetragonal phase, as well as the overall stability of the bulk Bi-2212 superconducting system, cannot withstand the presence of calcium oxide impurity in the Bi-2212 crystal structure.

Table 3. Variation of granularity degree parameters as a function of calcium oxide impurity addition amount and test forces applied.

	Applied Indentation Test Loads (N)				
Samples	***0.245***	***0.490***	***0.980***	***1.960***	***2.940***
	Relative volume fraction porosity (%)				
Pure	-----	1.7866	2.4975	2.7296	2.9443
Ca-1	1.86925	4.0223	5.3458	5.6894	5.9014
Ca-2	3.5660	6.8142	8.8896	9.1161	9.3306
Ca-3	6.7714	10.998	13.0505	13.4247	13.7361
Ca-4	9.2003	13.9441	16.5624	17.2358	17.3385
Ca-5	13.6252	18.4463	20.8858	21.3395	22.6452

IV. CONCLUSION

In this study, there seemed to be a strong link between the calcium oxide impurity addition mechanism and mechanical performances, durable tetragonal phase, and stabilization of granular $Bi_{2.1}Sr_{2.0}Ca_{1.1}Cu_{2.0}O_y$ crystal structure. Experimental values were determined from Vickers microindentation hardness tests conducted with various applied test loads ranging from 0.245 N to 2.940 N. Ca-doped Bi-2212 superconducting ceramic compounds were produced at different Ca molar concentrations using the solid-state reaction method. The observed Vickers microhardness curves allowed us to numerically determine parameters such as elastic moduli, shear modulus, and resilience of the Ca added Bi-2212 ceramic structure. It was observed that all mechanical performance parameters were strongly dependent on both the applied test loads and the presence of extra Ca-sites in the system. Thus, it is evident that calcium oxide impurity was successfully incorporated into the $Bi_{2.1}Sr_{2.0}Ca_{1.1}Cu_{2.0}O_y$ crystal structure. Furthermore, the presence of calcium impurity in the Bi-2212 ceramic system resulted in the increment in the crystal structure problems based on microvoids, strains, prevalent defects, bonding problems, and crystallinity faults based on interaction between adjacent layers of ceramics.

Therefore, the experimental results indicated that the pure sample exhibited much stronger mechanical performance parameters compared to the other Ca added Bi-2212 ceramics. Similarly, the Bi-2212 prepared with the maximum calcium impurity addition amount showed the highest response and sensitivity to the test forces applied due to its worst crystal quality.

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