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# Effect of annealing ambient conditions on crack formation mechanisms of bulk Bi-2212 ceramic systems

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## ABSTRACT

This study paves way to examine the influence of different annealing conditions (temperature range of 830-850°C and duration intervals 24-48 h) on the fundamental mechanical performance and characteristic quantities of polycrystalline $Bi_{2.1}Sr_{2.0}Ca_{1.1}Cu_{2.0}O_y$ (Bi-2212) superconducting ceramics by means of Vickers microindentation hardness tests at the various indentation test loads ($0.245 \leq F \leq 2.940$ N) and some available theoretical approaches. The annealing ambient plays an important role on the operable slip systems and crystal quality. The bulk Bi-2212 superconducting compound prepared at 840 °C and 24 h is found to be the least sensitive to the applied test load due to less structural problems, voids, cracks and stress raisers in the crystal system. Conversely, the excess annealing ambient complicates remarkably the control of crack growth size and velocity. Thus, relatively lower load can lead to the formation of crack and acceleration of crack rate up to the critical size and terminal velocity. The samples exhibit the typical indentation size effect (*ISE*) behavior as a result of predominant character of elastic recovery mechanism. As for the theoretical examination in the saturation limit regions, the indentation-induced cracking (*IIC*) model wins the comparison as it provides the most accurate results to the experimental findings.

## 1. Introduction

Mechanical performance of a material is in association with the response to the load and resulting deformation depending on the motions of internal omnipresent flaws, pores, voids, interior cracks and dislocations in the crystal system. Thus, the mechanical performance is one of the most important parameters affecting the economic service lifetime of materials. It is well known that the internal omnipresent flaws, dislocations and interior cracks are naturally introduced in the entire specimen during the basic processes: plastic deformation, solidification, and rapid cooling leading to the thermal stresses throughout the crystal structure. In case a load resulting in the irreversible deformation in the system is applied to the compound, 95% of deformation energy is dissipated as heat along the body. The remaining part of energy is stored internally, and the majority of energy retained is conserved into the strain energy for the internal omnipresent flaws, dislocations and interior cracks. Additionally, the flaws and dislocations move with the different difficulty level through the crystallographic directions and planes. Hence, the flaws and dislocations move along the slip system consisting of specific slip directions and planes. The inner term is related to the most closely packed (greatest linear density) directions with atoms when the latter one is attributed to the densest atomic packing (highest planar density) plane. The number of possible slip systems in the crystal matrix is a measure of plastic deformation. On this basis, the ceramic materials exhibit high brittleness nature due to intrinsic few active slip systems. In the current work, hardness test method is used to determine the general mechanical performance quantities.

It is well-known that hardness is received to be the resistance behavior against the localized plastic deformation. The hardness of a material can be determined from a number of quantitative hardness techniques where a small indenter leaves a notch on the specimen surface to be tested by using available applied load [1,2]. In the tests, the indentation depth sizes are measured under the controlled conditions of applied load, duration and rate of application [3,4]. The indentation leaves much deeper and larger notch size on the surface in case the material is relatively softer. Thus, the soft material exhibits lower hardness index number whereas the harder sample possesses higher one [5]. That is exactly why, the hardness is the most desired mechanical quantity for the ceramic compounds. Additionally, the researchers more frequently prefer to conduct the hardness measurements for the mechanical investigation of a material as compared to the other

mechanical tests due to their intrinsic rather simpler, cheaper, and less destructive/deformative test technique. In fact, that the other key mechanical quantities can be found with the aid of hardness index number determined [6] is another noteworthy advantage for the hardness tests. In the application fields, the most usage hardness tests can be mentioned as Rockwell, Brinell, Knoop and Vickers [4]. It is to be strongly emphasized here that considering the distinctive scales of the hardness methods, all the hardness results can easily be converted into each other. Among the test techniques mentioned above, the last two ones are referred as microindentation hardness tests due to the small indenter (notch) size grounded on relatively light load magnitudes applied on the specimen surface and provide superior performance for the measurement of small regions. Accordingly, for a ceramic compound (Bi-2212 ceramic products in the current study) the methods are totally useful for the investigation of mechanical performance quantities. With the detailed mechanical analyses, the researchers can easily decide whether the ceramic compound produced is useful for the heavy-industrial technology, advanced engineering, and large-scale application fields or not. On the other hand, it is received that the researchers have some difficulties to perform accurately the hardness measurements of brittle materials due to high susceptible to the cracking mechanisms based on the stress concentrations/amplifications, stress raisers and induced crack initiation (nucleation) sites in the crystal system.

Nowadays, the Bi-containing superconducting ceramic compounds with its three family members [7] are one of the most preferred materials in the heavy-industrial technology, advanced engineering, and large-scale application fields [8–11] due to their promising characteristic features: extremely higher critical temperature, thermodynamic stability, current density and magnetic field carrying capability [12,13]. Similarly, the easy preparation process, cheap contents, resistance to the humidity, and stability to the compositional contents and oxygen level, low power consumption and dissipations can be counted as the advantages of compounds [12–15].

In the present study, the effect of different annealing conditions including annealing temperature varying from 830°C to 850°C and annealing time between 24 h and 48 h on the fundamental mechanical performance (mechanical durability, fracture toughness and mechanical strength) and elastic and plastic deformations characteristic quantities of polycrystalline bulk Bi-2212 superconducting materials is examined with the aid of Vickers microindentation hardness tests at the various indentation test loads (0.245 N ≤ F ≤ 2.940 N) and some available theoretical approaches (Meyer's law, elastic/plastic deformation, proportional sample resistance model, modified proportional sample resistance, Hays–

Kendall and indentation-induced cracking model). Further, strong links are established between the annealing conditions and fundamental information about the formation of internal omnipresent flaws, stress amplification and consideration mechanism, durable tetragonal phase, crack propagation rate, operable slip systems, critical stress, elastic recovery, transgranular or intergranular fracture regions in the crystal system. According to the experimental results inferred from the present work, it is found that the preparation condition affects strongly the general mechanical quantities. The bulk Bi-2212 superconducting ceramic compound produced at 840°C for 24 h shows the highest mechanical performance features due to the formation of new operable slip systems in the Bi-2212 crystal matrix, leading to divert the mobility of internal omnipresent flaws, dislocations and interior cracks. Accordingly, the optimum annealing condition strengthens the mechanical strength, durability, stiffness, critical stress, and resistant against the failure by fatigue due to the improvement in the resistance toward to the crack propagation. In the discussion part of theoretical approaches, the hardness behavior of Bi-2212 superconducting material is also determined with respect to the applied test loads [16].

Shortly, in the remaining parts of paper, we will shed some lights on the subject matters in detail as follows:

- In Results and discussion part, differentiation of fundamental mechanical performance behaviors founded on new possible active slip systems in the Bi-2212 ceramic structure with the annealing conditions,
- In Part 3.1, mechanical modeling of load-independent vickers microindentation hardness values,
- In Section 3.2, examination of true Vickers microindentation hardness parameters with Meyer's law,
- In Part 3.3, view on mechanical characteristic features with proportional sample resistance model,
- In Section 3.4, examination of mechanical characteristic features grounded on modified proportional sample resistance model,
- In Part 3.5, elastic/plastic deformation model for examination of load-independent Vickers hardness parameters,
- In Section 3.6, examination of Vickers microindentation hardness with Hays–Kendall approach,
- In Part 3.7, research of load-independent micro-hardness parameters with indentation-induced cracking model,
- In Section 4, a brief review of main results is performed.

## 2. Experimental details for Bi-2212 ceramic compounds

The current study is a part of systematic investigation research grounded on the determination of best annealing condition (temperature between 830°C and 850°C with 10°C increment step and duration intervals 24–48 h with the enhancement of 24 h) for the fundamental characteristic quantities, viz. the superconducting, crystal quality, flux pinning mechanism, electrical, physical, microstructural, general mechanical performance and characteristic quantities. Previously, the detailed experimental procedures, high-purity chemical powders, heat-treatment methods, ceramic preparation technique, heating/cooling rates, characterization instruments, experimental test tools and related whole information are provided in Ref [17].

In the present study, it is indicated why the fundamental mechanical performance and characteristic quantities are forced to considerably change with the different annealing conditions by means of Vickers microindentation hardness test techniques in detail. Right after, the microhardness values obtained are thoroughly modeled by six standard theoretical methods: Meyer's law, elastic/plastic deformation, proportional sample resistance model, modified proportional sample resistance, Hays–Kendall and indentation-induced cracking approaches so that we provide the mechanical identification to the materials and examine the role of annealing ambient conditions on the hardness behavior (conventional indentation size effect, ISE and untypical reverse indentation size effect, RISE). As for the outline for the variation in the mechanical performance with annealing ambient conditions, we firstly determine the formation of permanent internal omnipresent flaws, pores, voids, defects, dislocations and interior cracks in the crystal structure depending on the annealing ambient conditions in the bulk Bi-2212 superconducting ceramic compounds. Thus, the structural problems mentioned above act as the stress raisers (playing much more significant role in the brittle ceramic materials) in the crystal matrix. In the presence of applied test load on the specimen surface, the stress raisers based on the orientation and geometry of local structural problems are much more amplified or concentrated. After then, the formation of cracks immediately begins from these problematic points, and the origins of cracks start to branchy move depending on the applied test load magnitude. Beyond the applied test load higher than the critical value, the cracks propagate rapidly toward to the critical velocity in crystalline ceramics. Once an advancing crack reaches to its critical size or terminal velocity, the failure and (transgranular or intergranular) fracture in the crystal system are inevitable. In other words, the difficulty increases for the control of crack growth size and velocity in case of the excess annealing temperature, and so the mechanical strength and fracture toughness are deleteriously affected. Also, the influence of annealing conditions on the variation of operable slip systems is discussed in the Bi-2212 crystal structure.

Experimentally, the Vickers hardness measurements are performed at the various indentation test loads ($0.245 \leq F \leq 2.940$ N) by the digital SHIMADZU HVM-2 model brand tester in the standard conditions. The measurements are taken from the different locations on the specimen surfaces for the duration of 10 s, and the sizes of two mutual notches are sensitively (within the accuracy of ±0.1 μm) recorded by means of the calibrated microscope. The related microhardness parameters are deduced from the equation of Table 2. Comparisons of Vickers microindentation hardness parameters in saturation limit regions to those of $H_{PSR}$, $H_{MPSR}$, $H_{EPD}$, $H_{HK}$ and $H_{IIC}$.

## 3. Results and discussion

Prior to the crucial discussions related to the mechanical performance results depending on the different annealing ambient conditions, the microhardness findings of all the bulk Bi-2212 superconducting materials against the applied test loads are obtained from Ref [17]. and inserted in the graphics (Figure 1) to investigate with the available approaches in detail. It is obvious from the figure that the annealing condition strongly affects the Vickers hardness values and does not change the general mechanical behavior (ISE) of compounds. Let us make much more phenomenological and sophisticated discussions on what happens in the crystal structure in the bulk Bi-2212 superconducting system with the differentiation in the annealing ambient conditions. The figure shows that the key mechanical performance quantities of compounds prepared are forced to change harshly. This is because; the deformation degree of a material (up to the load-independent saturation region) is remarkably dependent upon the annealing ambient conditions and especially magnitude of an applied test load. For the former effect, it is well known that any effect such as the preparation procedure, annealing ambient conditions, pressure, and dopant type leads to change remarkably the interatomic displacements and interatomic bonding forces in the crystal source. Accordingly, we should mention the formation of permanent internal omnipresent flaws, pores, voids, defects, dislocations and interior cracks in the crystal structure depending on the annealing ambient conditions in the bulk Bi-2212 superconducting ceramic compounds. In this regard, the experimental findings display that the combination of excess annealing temperature and time such as 850°C and 48 h supports the general structural problems in the crystal structure. Thus, the stress amplification/concentration grounded on
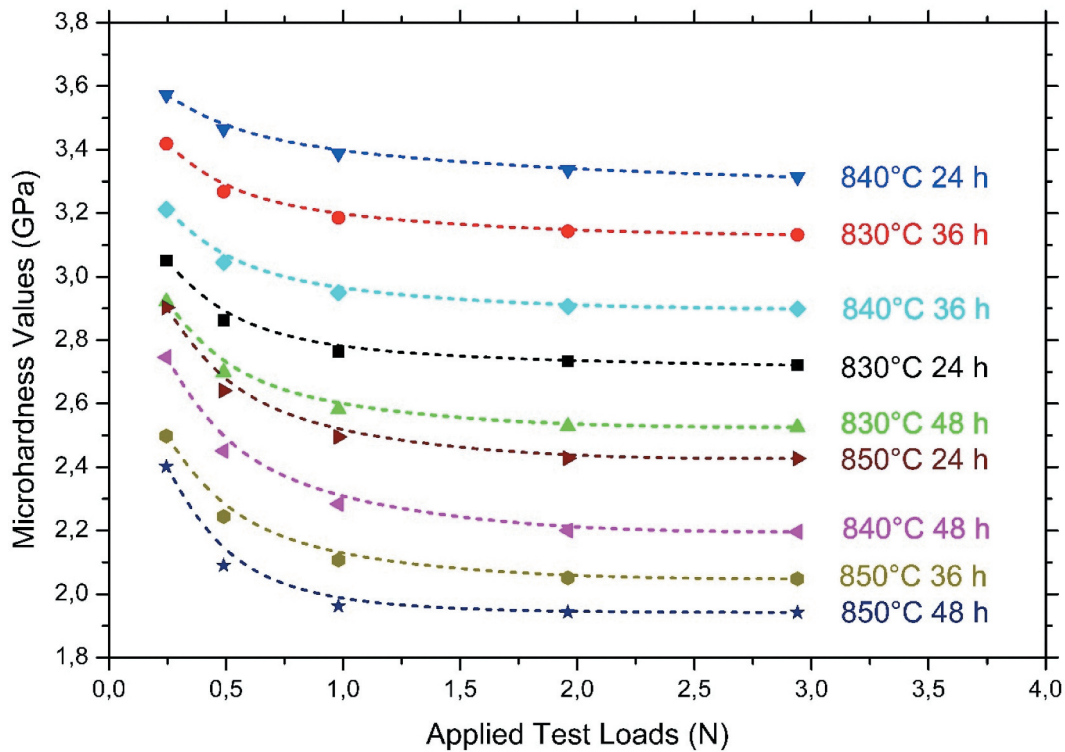
**Figure 1.** Variation of vickers microindentation hardness parameters over indentation test loads for all Bi-2212 superconducting ceramic compounds. (taken from Ref [17].

on the orientation and geometry of local structural problems mentioned above is much more easily supplied in the crystal system and the fracture strength belonging to the bulk Bi-2212 material is harshly damaged. Shortly, it is customary to confirm that the formation of flaws and cracks (due to the excess annealing ambient conditions) with their superior ability to the amplification and concentration of stress applied serves as the stress raiser mechanism (playing much more significant role in the brittle ceramic materials) in the crystal matrix. In other words, we can discuss that the excess annealing ambient conditions triggers the formation probability of permanent structural problems and especially internal omnipresent flaws possessing the capability for the nucleation of cracks throughout the crystal structure. It is obvious that the fracture strength of polycrystalline Bi-2212 superconducting material is strongly dependent upon the preparation conditions. In case of the excess annealing ambient conditions, the rate of crack acceleration can easily be enhanced with the increment in the applied test load. Conversely, the optimum annealing ambient conditions seem to eliminate much more structural problems and corresponded stress raisers in the crystal structure, and in fact clearly strengthen and regulate the general mechanical performance features of bulk Bi-2212 superconducting material. All in all, the Bi-2212 superconducting ceramic compound prepared at 840°C for 24 h supplies the desirable combinations of mechanical performance and characteristic features. The facts have already been supported by the dc

electrical resistivity measurement results directly related to the microstructure problems in the crystal system [18]. Namely, the Bi-2212 material prepared at the optimum annealing ambient conditions exhibits the smallest normal state resistivity at room temperature (14.37 mΩcm), residual resistivity (7.87 mΩcm), $\rho_{100K}$ (11.16 mΩcm), $\rho_{norm}$ (3.48) and the maximum residual resistivity value ratio of 1.29. On the other hand, in case of the 850°C and 48 h annealing ambient condition, the fundamental electrical characteristics reach to their maximum points such as 41.56 mΩcm for the normal state resistivity at room temperature, 39.39 mΩcm for residual resistivity, 40.45 mΩcm for $\rho_{100K}$, 36.44 for $\rho_{norm}$ and deepest point of 1.03. It was concluded that the improvement/degradation of electrical parameters stemmed from the enhancement/decrement in the number of mobile hole carrier concentrations, lattice strain, impurity scattering, interaction between the superconducting grains and systematic structural problems in the crystal structure. Similarly, the former compound possesses the highest onset/offset critical transition temperature values of 83.41 K/81.16 K whereas the latter sample presents the global minimum onset/offset values of 74.63 K/ 53.85 K. The parameters related to the broadening degree are observed to be about 2.25 K and 20.78 K for the best and worst sample, respectively. The main changes in the characteristic superconducting quantities were attributed to the positive/negative effects on the active and effective electron-phonon coupling probabilities in the homogeneous superconducting

cluster percentages in the paths, amplitude of pair wave function, polaronic effect in the polarizable lattices, overlapping of Cu-3d and O-2p wave functions, homogeneities in the oxidation states and densities of electronic states at the Fermi energy level [18].

It is another probable result inferred from the Vickers microhardness tests that the optimum (excess) annealing ambient conditions may induce (limit) the operable slip systems in the Bi-2212 crystal lattice. Correspondingly, it can be put forward that in case of excess annealing ambient conditions, the propagation of cracks rapidly reaches to the critical velocity along the grain boundaries and is not decelerated easily under relatively lower test load due to the induced stress amplification and consideration mechanism. Thus, the fracture cracks pass through the intergranular regions. Conversely, the material with stronger fracture toughness and mechanical strength does not allow the rate of cracks accelerate up to the critical size and terminal velocity. Accordingly, the compound can tolerate much more applied force, and the fracture for the cracks passes along with the grains (transgranular regions).

## 3.1. Mechanical modeling of load-independent vickers microindentation hardness values

As it is declared in the introduction part, the load-independent Vickers microindentation hardness parameters of bulk polycrystalline Bi-2212 ceramic material in the plateau limit regions are inspected by means of the valuable mechanical modeling approaches such as Meyer's law, elastic/plastic deformation, proportional sample resistance model, modified proportional sample resistance, Hays–Kendall and indentation-induced cracking models. Now, the results of models listed above will in turn be looked at and the related important comments will be made.

## 3.2. Examination of true vickers microindentation hardness parameters with Meyer's law

Meyer's law is known to be a selected valuable model that enables us to scrutinize the general mechanical characterization (received as ISE: typical decrement in the Vickers microhardness parameters with the applied indentation test loads, and RISE: unusual increment in the Vickers microhardness parameters with the applied indentation test loads) of material under external stress. In terms of Meyer's law (provided below in detail), the variation of microindentation test load (F) is quantitatively determined by a Meyer constant and exponential power of $n$ for the indenter diagonal size [19]:

$$F = A_{Meyer}d^n \tag{1}$$

in the equation, the abbreviation of $A_{Meyer}$ shows the constant microhardness value for the material when the power of $n$ shows Meyer number under applied test load. Meyer number obtained becomes a leader for defining the mechanical characteristic behavior of compound as using a stress applied on the surface. In this context, the number of $n$ with three varied scales takes the role of decision-making authority. Namely, number 2 for the $n$ value is important to determine the general mechanical characteristic of material exhibiting ISE or RISE behavior. If a material when under the test load has the $n$ parameter smaller than 2 (n < 2), it is customary to say that the material studied shows the standard ISE behavior. If $n>$ is higher than the value of 2, the compound exhibits the unconventional RISE feature. If n = 2, the microindentation hardness parameters are independent upon the applied test load [20]. The main difference between the ISE and RISE characteristic behavior is the recovery mechanism. That is, for the compound exhibiting the ISE nature (predominant character of elastic recovery) the elastic and plastic deformations appear instantaneously whereas the permanent and non-recoverable deformations are observed due to the presence of crack propagation. It is to be reminded here that the materials exposed to the mechanical test load exhibit either elastic or plastic deformation or together in the crystal system. The former deformation related to the proportional variation of stress and strain under the applied load is nonpermanent. Whenever the applied load is removed from the specimen surface, the material returns into its original situation. On the other hand, the latter one is permanent deformation and is the measurement of strength and hardness for a compound. The slip systems (combination of slip direction and slip plane) play an active role in the plastic (irreversible = permanent change of shape) deformation.

One can see the variation of LnF versus Lnd graphics for all the Bi-2212 materials in Figure 2. After using the data extrapolation method on the results, we numerically give every curve slope (regarding the $n$ value) and crossing point of y-axis ($A_{Meyer}$ parameter) belonging to the bulk compounds in Table 1. It is obvious from the table that all the extrapolated parameters are found to be smaller than 2. This is in association with the fact that all the compounds present the typical ISE behavior (with elastic recovery mechanism) even if prepared at different annealing conditions [21]. The $n$ value is calculated to be in a range of 1.848–1.942 for the Bi-2212 material prepared at the annealing temperature of 840°C for the duration of 24 h (best) and 850°C for 48 h (worst), respectively. The model also verifies that the optimum annealing ambient condition is the combination of 840°C and 24 h for producing the bulk Bi-2212 superconducting material with superior mechanical performance behaviors due to less
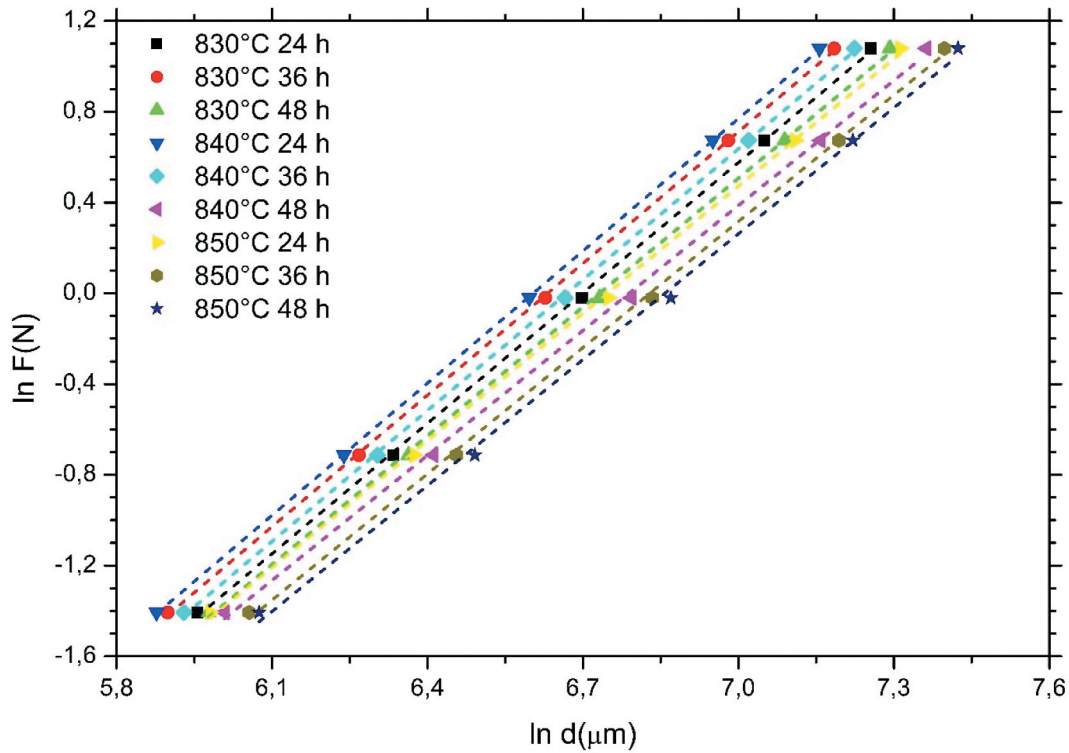
**Figure 2.** Variation of LnF versus Lnd graphics for all bulk polycrystalline Bi-2212 materials.

**Table 1.** Microhardness characteristic parameters deduced from 6 different theoretical models for all bulk Bi-2212 superconducting ceramic compounds.

| | Meyer's Law | | PSR Model | | | MPSR Model | | EPD Model | | HK Model | IIC Model | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Samples | $A_{MEYER} \times 10^{-6}$ (N/μm²) | n | $\alpha \times 10^{-4}$ (N/μm) | $\beta \times 10^{-6}$ (N/μm²) | $W \times 10^{-2}$ (N) | $A_{0MPSR} \times 10^{-5}$ (N/μm) | $A_{1MPSR} \times 10^{-6}$ (N/μm²) | $d_e \times 10^{-1}$ (μm) | $A_{2EPD} \times 10^{-2}$ (N$^{1/2}$/μm) | $W \times 10^{-1}$ (N) | $A_{3HK} \times 10^{-6}$ (N/μm²) | $K \times 10^{-6}$ (N$^{(3-5mA)/3}$/μm$^{(2-3m)}$) | m |
| 830°C 24 h | 2.680 | 1.915 | 0.821 | 1.404 | 3.434 | −1.781 | 1.463 | 0.331 | 0.119 | 0.275 | 1.453 | 1470.695 | −0.376 |
| 830°C 36 h | 2.700 | 1.933 | 0.715 | 1.631 | 2.106 | 0.657 | 1.672 | 0.270 | 0.128 | 0.244 | 1.675 | 12,089.596 | −0.281 |
| 830°C 48 h | 2.965 | 1.891 | 1.031 | 1.284 | 4.997 | −3.709 | 1.363 | 0.429 | 0.113 | 0.353 | 1.343 | 43.983 | −0.545 |
| 840°C 24 h | 2.647 | 1.942 | 0.690 | 1.733 | 0.006 | 6.807 | 1.734 | 0.254 | 0.132 | 0.241 | 1.774 | 28,244.120 | −0.242 |
| 840°C 36 h | 2.716 | 1.922 | 0.799 | 1.499 | 0.030 | −1.007 | 1.554 | 0.313 | 0.123 | 0.265 | 1.548 | 2816.980 | −0.348 |
| 840°C 48 h | 3.883 | 1.835 | 1.501 | 1.079 | 6.574 | −2.519 | 1.172 | 0.660 | 0.104 | 0.552 | 1.160 | 6.191 | −0.631 |
| 850°C 24 h | 3.402 | 1.866 | 1.250 | 1.216 | 6.152 | −4.553 | 1.310 | 0.527 | 0.110 | 0.433 | 1.287 | 2.004 | −0.695 |
| 850°C 36 h | 3.185 | 1.853 | 1.253 | 1.019 | 6.737 | −4.765 | 1.107 | 0.573 | 0.101 | 0.467 | 1.085 | 3.379 | −0.653 |
| 850°C 48 h | 3.126 | 1.848 | 1.163 | 0.967 | 9.512 | −12.422 | 1.087 | 0.543 | 0.099 | 0.398 | 1.030 | 23,866.965 | −0.722 |

structural problems, voids, cracks and stress raisers in the crystal system. Moreover, the constant microhardness value of $A_{Meyer}$ parameter shows that the bulk Bi-2212 ceramic compound produced at 840°C for 24 is found to be the least sensitive to the applied test load. It is another probable result deduced from the table that once the applied load is larger than the critical stress value, the fracture occurs in the transgranular regions for the sample. On the other hand, the sample prepared at the excess annealing ambient condition (850°C for 48 h), the fracture predominantly appears throughout the intergranular regions.

### 3.3. View on mechanical characteristic features with proportional sample resistance model

Secondly, we inspect the effect of different annealing temperature and time on the general mechanical characterization of solid Bi-2212 superconducting ceramic materials by means of proportional sample resistance

(PSR) approach [22]. Besides, here we point out the dependence of internal omnipresent flaws, dislocation density, mobility of dislocations, stress amplification and consideration mechanism, slip systems, cracks and dislocation motion in the material on the annealing ambient condition and applied test load. In the current model, the energy disperses along with the structural problems, internal omnipresent flaws, pores, voids, defects, dislocations and interior cracks. That is exactly why, the surface energy parameter becomes a leader to define the general mechanical characterization belonging to the bulk materials. The related formulation for the theoretical approach consists of two continuing parts founded on the reversible and irreversible deformation [22], viz. the first one is the surface energy parameter (shown as α in the equation given below) and the last one is the microindentation hardness constant (abbreviated as β) [23]. If a material possesses positive surface energy parameter, the elastic recovery mechanism (production of

**Table 2.** Comparisons of vickers microindentation hardness parameters in saturation limit regions to those of $H_{PSR}$, $H_{MPSR}$, $H_{EPD}$, $H_{HK}$ and $H_{IIC}$.

| Samples | $H_{PSR}$ (GPa) | $H_{MPSR}$ (GPa) | $H_{EPD}$ (GPa) | $H_{HK}$ (GPa) | $H_{IIC}$ (GPa) | Hv (GPa) |
|---|---|---|---|---|---|---|
| *830℃ 24 h* | 2.604 | 2.713 | 2.626 | 2.694 | 2.824 | 3.050–2.721 |
| *830℃ 36 h* | 3.025 | 3.101 | 3.038 | 3.106 | 3.185 | 3.419–3.132 |
| *830℃ 48 h* | 2.381 | 2.528 | 2.368 | 2.490 | 2.773 | 2.921–2.525 |
| *840℃ 24 h* | 3.214 | 3.216 | 3.231 | 3.290 | 3.422 | 3.573–3.314 |
| *840℃ 36 h* | 2.780 | 2.882 | 2.806 | 2.871 | 2.985 | 3.212–2.898 |
| *840℃ 48 h* | 2.001 | 2.173 | 2.006 | 2.151 | 2.466 | 2.746–2.196 |
| *850℃ 24 h* | 2.255 | 2.429 | 2.244 | 2.387 | 2.695 | 2.903–2.427 |
| *850℃ 36 h* | 1.890 | 2.053 | 1.892 | 2.012 | 2.426 | 2.498–2.048 |
| *850℃ 48 h* | 1.793 | 2.016 | 1.817 | 1.910 | 1.869 | 2.402–1.942 |

both elastic and plastic deformations with the applied load) plays a predominant role in the crystal structure. Namely, the compound exhibits the standard *ISE* nature. Conversely, the sample shows the untypical *RISE* behavior in case of negative surface energy parameter. In this respect, the model is perfect to define the mechanical identification of the material that exhibits the *ISE* or *RISE* behavior. Thus, the model works perfectly to describe the mechanical identification of the material exhibiting the *ISE* nature with the positive surface energy value. As for the formulization,

$$F = ad + \beta d^2 \qquad (2)$$

As a result of division of applied test load with notch size, we display the graphics related to the variation of *F/d* over *d* for all the bulk Bi-2212 superconducting ceramic compounds in Figure 3 to calculate the *a* and *β* parameters form the data extrapolation method. One can see all the computations in Table 1. It is clear from the table that ever sample prepared exhibit the

typical *ISE* feature due to positively calculated values of *a*. It is normally to confirm that the elastic recovery mechanism (production of both reversible and reversible deformations with the applied load) plays an important role in the material characterization. Numerically, the minimum *a* constant is found to be about $0.69 \times 10^{-4}$ N/μm for the bulk Bi-2212 ceramic material prepared at the annealing temperature of 840℃ for the duration of 24 h. Similar to the discussions of Meyer's law, the PSR model presents that the sample with much more crystal quality possesses the highest mechanical performance quantities. In this respect, the best annealing ambient conditions mentioned above block strongly to produce the structural problems, internal omnipresent flaws, pores, voids, defects, dislocations and interior cracks (leading to formation of the stress raisers and concentration region) in the crystal system. Thus, relatively rather more applied load can damage the material. Even, the calculated microindentation hardness constant of
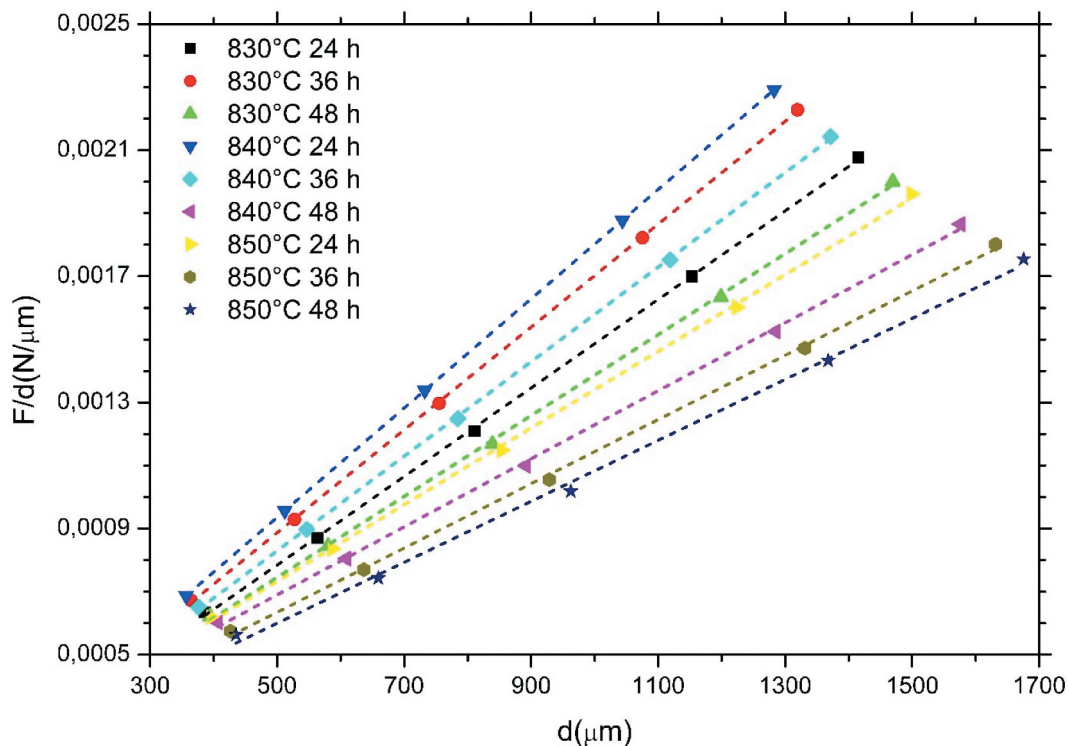


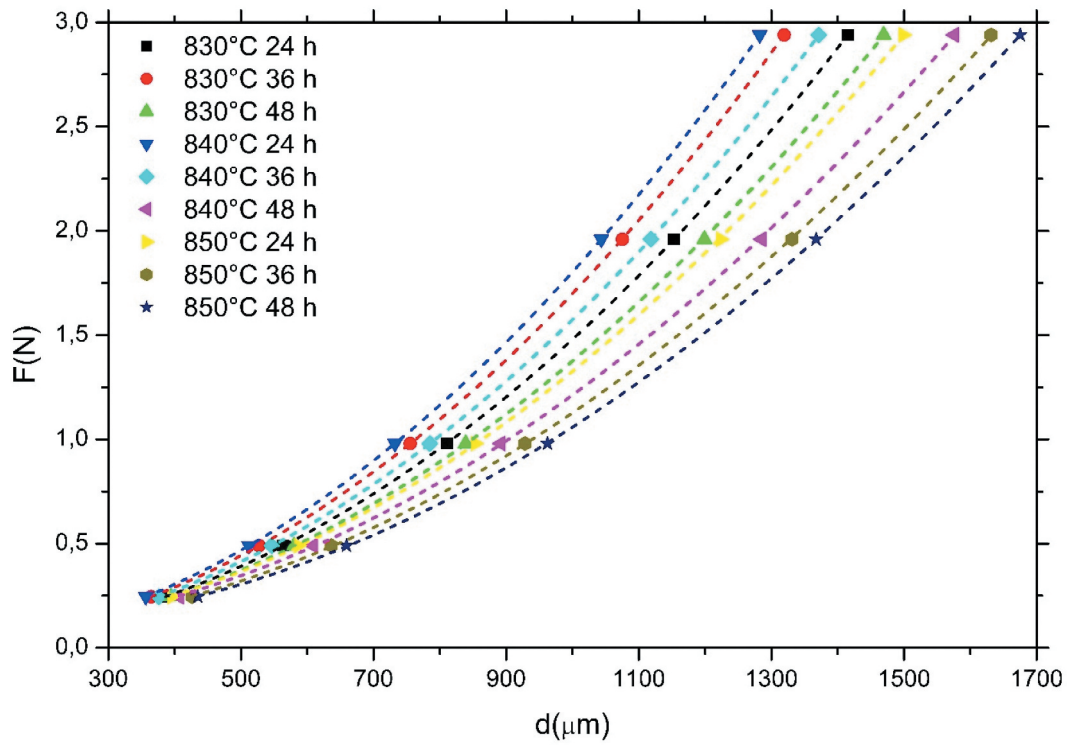**Figure 3.** Change of *F/d* over *d* for all bulk Bi-2212 superconducting ceramic compounds.

**Figure 4.** Linear plots of applied load *F* over indentation length *d* for all samples.

*β* already confirms the findings/facts discussed above. It is seen from the table that the bulk Bi-2212 superconducting sample prepared by the best (worst) annealing ambient conditions obtain the maximum (minimum) *β* value of 1.733 (0.967) $\times 10^{-6}$ N/μm². The values reveal that the excess annealing temperature and time damage the fundamental mechanical performance quantities.

Unlike the Meyer's Law, this model also provides an opportunity (free, simple, fast, practical and reliable) us to compare the load-independent Vickers microindentation parameters using the following relation [24]:

$$H_{PSR} = 1854.4\beta \qquad (3)$$

All the true Vickers microindentation parameters obtained are numerically inserted in Table 2. It is apparent from the table that the results deduced from the PSR model in the plateau regions are found to be slightly lower than the real ones for all the materials. On this basis, the theoretical PSR model can easily be used to search the general mechanical characteristic behavior of bulk Bi-2212 superconducting ceramic systems. Similarly, the model with the main findings enables us to discuss the crystal structure quality of material.

### 3.4. Examination of mechanical characteristic features grounded on modified proportional sample resistance model

Modified proportional sample resistance (*MPSR*) model is another theoretical approach for the examination of the effect of different annealing temperature and time

on the general mechanical characterization of bulk Bi-2212 superconducting materials. It is of course that the model enables us to explain the variation of fundamental mechanical performance quantities of the compounds depending on the annealing ambient conditions. When compared to the *PSR* approach, the *MPSR* model includes an extra constant abbreviated as $W_{MPSR}$ related to the smallest applied stress for the formation of indenter diagonal length on the specimen surface. Namely,

$$F = W_{MPSR} + A_{0MPSR}d + A_{1MPSR}d^2 \qquad (4)$$

where $A_{0MPSR}$ and $A_{1MPSR}$ parameters illustrate the surface energy and microindentation hardness constants grounded on the dissipation energies due to the irreversible deformation of a unit volume. One can see the differentiation of varied test loads over the indentation notch size (variation of *F* against *d*) in Figure 4 in detail. Like to the sister model of PSR approach, $W_{MPSR}$, $A_{0MPSR}$ and $A_{1MPSR}$ parameters inferred from the graphs with the aid of the data extrapolation method are numerically provided in Table 1. According to the numerical values, all the materials have the positive $A_{1MPSR}$ parameters intervals 1.734–1.087 $\times 10^{-6}$ N/μm². The maximum value (least sensitive to the applied load) ascribes to the material produced at the best annealing ambient condition whereas the minimum parameter (most sensitive to the applied load) is observed for the material prepared at the worst annealing ambient condition. This shows that in the present work every compound prepared exhibits the typical *ISE* behavior but with the various capacities. In this

respect, the sample produced at the best annealing ambient condition displays the highest *ISE* characteristic nature. As for the calculated $W_{MPSR}$ values, all the Bi-2212 superconducting samples show the parameters in the different sizes. The best sample is found to exhibit the highest mechanical durability, mechanical strength and fracture toughness against to the applied test load. Moreover, it is normal to establish a link between the model findings and structural problems grounded on the annealing ambient conditions. The model also supports the discussions performed on the mechanical performance quantities in the PSR model part.

Similar to the PSR approach, we compare the original experimental microindentation Vickers hardness parameters to the calculations based on the MPSR model that uses the relation given below:

$$H_{MPSR} = 1854.4A_{1MPSR} \tag{5}$$

One can see all the computations in Table 2 in detail. It is apparent from the table that the computations of *MPSR* model are found to be very very slightly lower than the experimental values in the saturation limit regions. Thus, this model is also useful to investigate the mechanical performance and characterization quantities of bulk Bi-2212 superconducting ceramic compound. In fact, if a researcher is forced to choose the theoretical model between PSR and MPSR approaches, it seems that the MPSR model is more suitable for the investigation of mechanical characteristics belonging to the bulk Bi-2212 compounds.

### 3.5. Elastic/plastic deformation model for examination of load-independent vickers hardness parameters

A researcher can use the elastic/plastic deformation (EPD) model to decide whether the material exhibits the ISE or RISE mechanical characteristic feature grounded on the elastic recovery mechanism. In fact, the model can much more be preferred in case of the predominant character of inelastic deformation in the system. This is because, the EPD method includes the extra new term (related to the plastic deformation) as a correlation factor in the theoretical formula so that the plastic deformation is emphasized. On this basis, the impression diagonal length within the plastic deformation can be evaluated from the relation [25]:

$$F = A_{2EPD}(d_e + d_p)^2 \tag{6}$$

here we experimentally measure the $d_p$ values as a function of applied test load ($F$). Accordingly, $A_{2EPD}$ and $d_e$ parameters are found from the variation of $F^{1/2}$ as a function of $d_p$ graphs using the data extrapolation method as depicted in Figure 5. It is to be stressed here that the sign of $d_e$ parameter defines the mechanical characteristic nature of material studied. One can observe all the $A_{2EPD}$ and $d_e$ computations obtained in Table 1. It is obvious from the table that every $d_e$ parameter is calculated to be positive within the different values due to the different *ISE* characteristic nature. In this respect, all the materials studied show the usual *ISE* behavior to the applied test load. As expected that the bulk Bi-2212 superconducting material prepared at the best annealing ambient conditions presents the strongest ISE feature depending on the smallest $d_e$ value of 0.0254 μm. This is related to the crystallinity quality and response to the applied field, having already discussed in the previous models. Furthermore, the same sample has the largest $A_{2EPD}$ parameter. This means that the best sample is noted to show the highest mechanical durability, mechanical strength and fracture toughness against to the applied test load. In fact, it is not wrong to point out that in the case of the high applied load the mechanical fracture occurs predominantly in the transcrystalline regions instead of throughout the intergranular regions. As for the comparison of the data with each other to determine the availability of model to the load-independent microhardness parameters, we calculate the $H_{2EPD}$ values using the following equation:

$$H_{EPD} = 1854.4A_{2EPD} \tag{7}$$

One can see the related computation value in Table 2. It is visible from the table that the value obtained from the EPD theoretical approach is found to be between those of PRS and MPRS approaches in the plateau regions. On this basis, it can be confirmed that the EPD theoretical method is a useful approach to describe the true Vickers microindentation hardness parameters of bulk Bi-2212 superconducting materials all in all, the EPD model is the practical way to anticipate the fundamental mechanical performance and characteristic quantities of Bi-2212 superconducting materials under the applied indentation test loads.

### 3.6. Examination of vickers microindentation hardness with Hays–Kendall Approach

In this part of the paper, we theoretically search the fundamental mechanical performance and characteristic quantities belonging to the bulk polycrystalline Bi-2212 superconducting ceramic compounds with the assistant of the Hays–Kendall (*HK*) approach [26]. This model is especially preferred for the sample exhibiting the *ISE* behavior due to its inherit formula. Namely, in the model there is a critic applied test load value (abbreviated as *W* that reveals the material exhibiting the *ISE* or *RISE* behavior depending on the positive or negative value) beyond which the irreversible deformation appears immediately on the specimen surface due to the deeply penetration of indenter into the bulk form of sample [27]. Hence, the differentiation in the indentation impression size as a function of applied
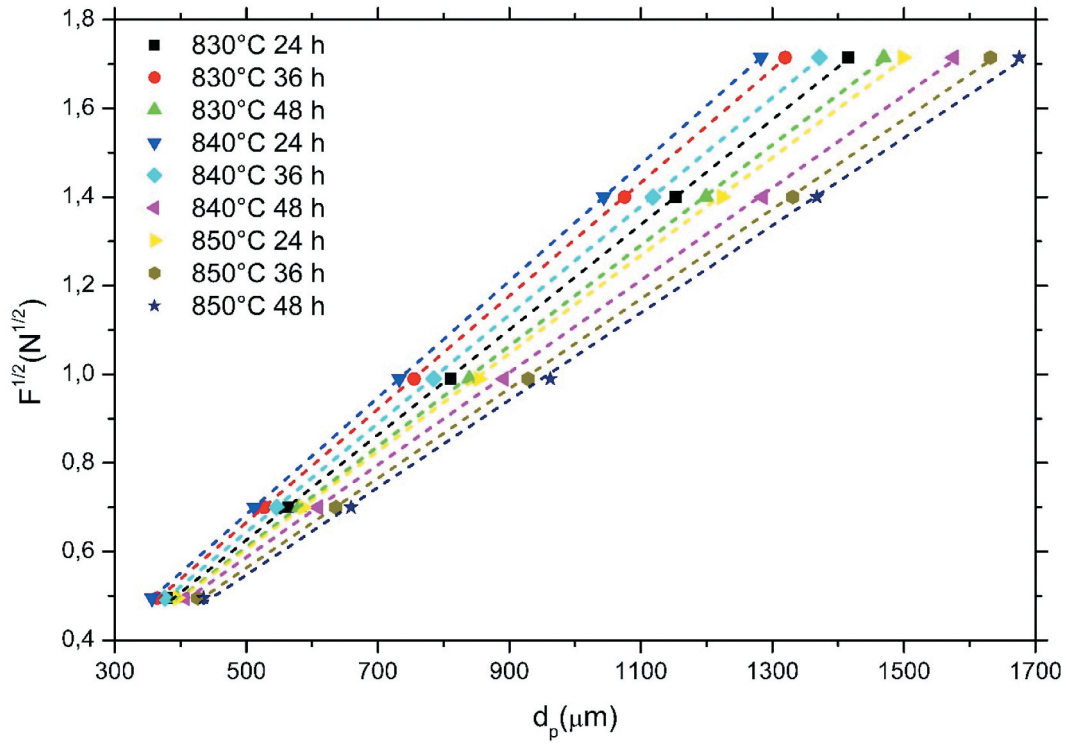
**Figure 5.** Plots of applied test load $F^{1/2}$ over plastic deformation $d_p$ for Bi-2212 ceramic materials.

test load is formalized by the effective load, $F_{eff} = F - W$. Namely,

$$F - W = A_{3HK}d^2 \quad (8)$$

here the parameter of $A_3$ demonstrates the microindentation hardness constant and the $W$ shows the indentation test load. We experimentally measure the $d$ values over the applied test load ($F$) from Figure 6 to obtain the constants of $A_{3HK}$ and $W$ parameters using the data extrapolation method. The constants computed are numerically listed in Table 1. It is visible from the table that all the Bi-2212 superconducting materials prepared in this work present the *ISE* nature as a result of the positive $W$ value. Thus, it is to be mentioned here that the material characterization is defined by the elastic recovery mechanism for all the compounds. According to the table, the bulk Bi-2212 superconducting compound prepared at he best annealing ambient shows the smallest $W$ value of 0.0241 N that means the least sensitive to the applied test load. Besides, the formula indicates that the smaller $W$ value a material has, the larger $A_{3HK}$ value it exhibits. Hence, as expected, the same sample possesses the maximum $A_{3HK}$ value of 1.774 (x10$^{-6}$ N/µm$^2$). Accordingly, the HK approach verifies that the compound presents the largest mechanical durability, mechanical strength and fracture toughness over the applied test load due to the highest crystal quality with the least stress raisers and concentration regions in the crystal matrix. That is, much more applied load needs to damage the specimen surface. In this regard,

it is not wrong to say that the annealing ambient condition is the determinant factor for the formation of crack and acceleration of crack rate up to the critical size and terminal velocity under the test load. We also analyze the load-independent Vickers hardness values in the saturation limit region with the aid of following equation:

$$H_{HK} = 1854.4A_{3HK} \quad (9)$$

$H_{3HK}$ values computed are numerically tabulated in Table 2. According to the table, all the data are found to be slightly lower than the original Vickers hardness values in the saturation limit regions. Thus, the HK model is thought to be one of the best theoretical models to examine the fundamental mechanical performance and characteristic quantities of bulk polycrystalline Bi-2212 superconducting ceramic compounds.

## 3.7. Research of load-independent microhardness parameters with indentation-induced cracking model

The indentation-induced cracking (*IIC*) method constitutes the last theoretical model to inspect the mechanical performance and characterization of bulk polycrystalline Bi-2212 ceramic materials [27–29]. Thus, the method is useful approach to decide what behavior (ISE or RISE) the material presents. In the model four main factors are explained for the resistance toward the notch diagonal size. These are (I) indenter friction; (II) reversible deformation; (III)
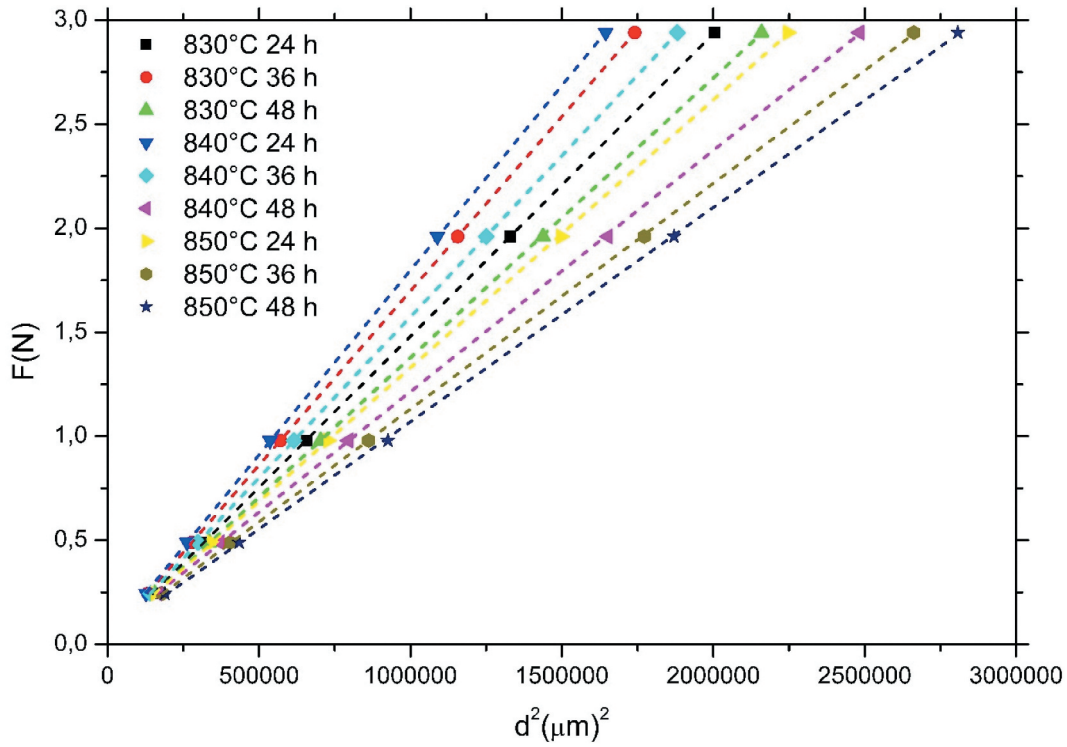
**Figure 6.** Linear graphs of applied indentation load $F$ against indentation notch length $d^2$ for every compound.

irreversible deformation and (IV) crack mechanism. One can see the *IIC* model formulation below:

$$H_{IIC} = \lambda_1 K_1 \left(F/d^2\right) + K_2 \left(F^{5/3}/d^3\right) \quad (10)$$

in the relation the abbreviation of $\lambda_1$ displays the constant for the material type. When $K_1$ reveals the geometry of indenter, $K_2$ provides the test load applied. As received, in case the material studied shows the perfect brittle nature (like our samples) the constant of $\lambda_1$ goes to 0. Accordingly, the relation is obtained to be $K_2$ $(F^{5/3}/d^3)$, and the last form of new equation is provided below:

$$H_{IIC} = K \left(\frac{F^{5/3}}{d^3}\right)^m \quad (11)$$

in the relation, the $K$ and $m$ parameters are related to the original hardness constants and evaluated from the data extrapolation method on the variation of ln $(Hv)$ versus indentation test load, $\ln(F^{5/3}/d^3)$ curves as depicted in Figure 7. All the calculation results are listed in Table 1 in detail. Similar to the Meyer's Law, in the *IIC* model $m$ has a special value such as 0.6 beneath which the superconducting compound obeys the standard *ISE* behavior. On the other hand, from the $m$ value of 0.6 onwards the bulk Bi-2212 superconducting material exhibits the unusual RISE behavior. For our study, all the materials produced at different annealing ambient conditions possess the $m$ values smaller than the critical value of 0.6. In this context, every material shows the typical *ISE* behavior. further, the table reveals that the bulk Bi-2212

sample prepared at the best annealing ambient conditions illustrates the maximum *ISE* nature with the highest mechanical strength and fracture toughness to the indentation test loads. Besides, it can be argued that much more applied test load magnitude needs for the formation of crack and acceleration of crack rate up to the critical size and terminal velocity in the sample. Thus, under enough applied load the fracture occurs in the transgranular regions. We also survey the true Vickers hardness ($H_{IIC}$) values in the plateau limit region. One can encounter all the $H_{IIC}$ computations in Table 2. Based on the table results, all the calculations are found to be perfectly closer to the load-independent Vickers microindentation hardness values in the saturation limit regions. It is just this last descriptor that confirms the most successful method to scrutinize the mechanical performance and characterization of bulk polycrystalline Bi-2212 ceramic materials.

## 4. Conclusion

In the present work, we establish a strong link between the annealing ambient conditions and fundamental mechanical performance and characteristic properties of bulk polycrystalline $Bi_{2.1}Sr_{2.0}Ca_{1.1}Cu_{2.0}O_y$ superconducting ceramics with the assistant of Vickers microindentation hardness measurements performed at the different indentation test loads between 0.245 N and 2.940 N and available theoretical modeling approaches, namely, Meyer's law, elastic/plastic deformation, proportional sample resistance model,
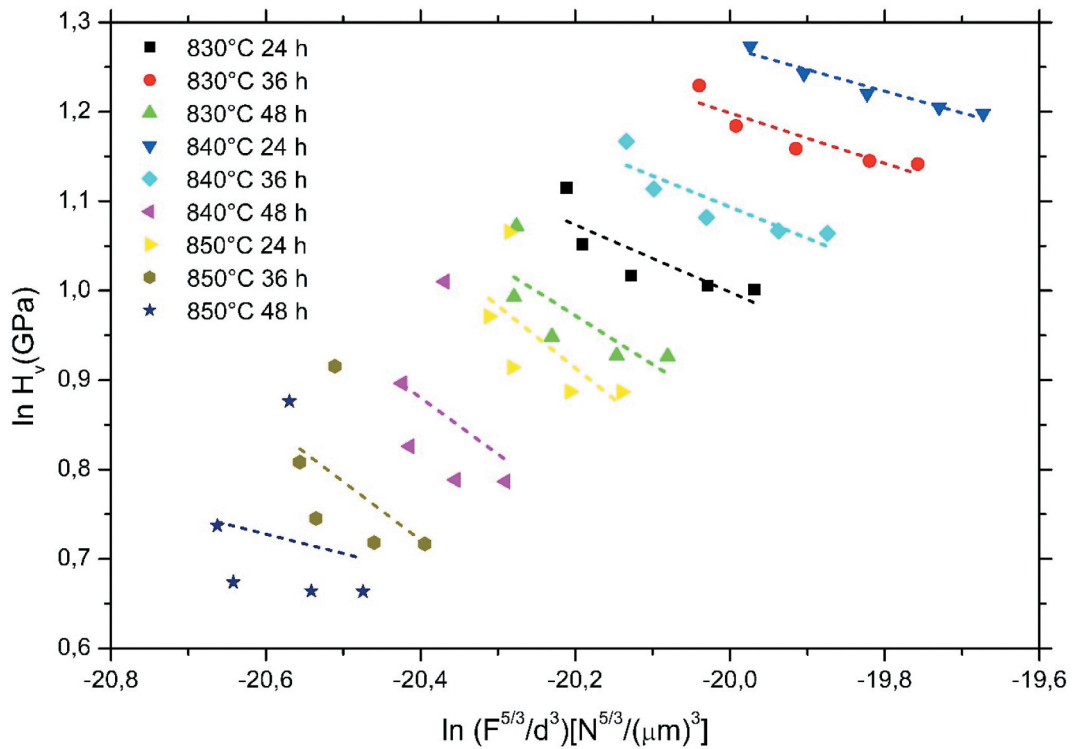
**Figure 7.** Variation of ln(*Hv*) versus indentation test load, ln($F^{5/3}/d^3$) curves for all materials prepared.

modified proportional sample resistance, Hays–Kendall and indentation-induced cracking model in the literature. It is found that the fundamental mechanical performance and characteristic quantities are strongly dependent upon the annealing ambient conditions due the formation of permanent internal omnipresent flaws, pores, voids, defects, dislocations and interior cracks (leading to the stress raisers to be concentrated or amplified by the applied test load) in the crystal structure. Considering all the findings mentioned, the differentiation of mechanical performance and characteristic behaviors, and formation of possible operable slip systems, internal omnipresent flaws, stress amplification and consideration mechanism based on the annealing ambient conditions are as follows:

- The Vickers microindentation hardness measurement results indicate that the optimum annealing condition is observed to be the combination of 840°C annealing temperature and 24 h annealing duration for preparing the Bi-2212 ceramic material with the least structural problems, voids, cracks, stress raisers and concentration region in the crystal system. On the other hand, the excess annealing ambient conditions are the driving force for the increase of stress raisers (capable of amplifying an applied test load) and induced crack nucleation sites in the Bi-2212 crystal system.
- The sample prepared at the best annealing conditions can tolerate much more applied force within itself while the compounds produced at

the excess annealing ambient conditions cannot withstand the load and the formation of crack begins depending on the stress raisers and stress concentrations/amplifications even under lower applied test load. In other words, the stress amplification/concentration grounded on the orientation and geometry of local structural problems mentioned above is much more easily supplied in the Bi-2212 sample prepared at the excess annealing ambient conditions and the fracture strength belonging to the bulk Bi-2212 material is harshly damaged. In fact, the rate of cracks accelerates to rapidly the critical size and terminal velocity; hence the mechanical fracture occurs through the intergranular regions. It is to be emphasized here that the difficulty increases for the control of crack growth size and velocity, and accordingly the mechanical strength and fracture toughness are deleteriously affected.

- In case of the production at the best annealing ambient conditions, much more applied test load magnitude needs for the formation of crack and acceleration of crack rate up to the critical size and terminal velocity in the sample. Thus, under enough applied load the fracture occurs in the transgranular regions. It seems obviously that the optimum annealing ambient conditions seem to eliminate much more structural problems and corresponded stress raisers in the crystal structure, and in fact clearly strengthen and regulate the general mechanical performance features of

bulk Bi-2212 superconducting material. To sum up, the annealing ambient is the determinant factor for the formation of crack and acceleration of crack rate up to the critical size and terminal velocity under the test load.

- Additionally, the model examination findings show that every compound presents the standard indentation size effect behavior (predominant character of elastic recovery mechanism) but with the different capacities. On this basis, the best sample possesses the highest *ISE* characteristic nature due to its superior mechanical strength and fracture toughness features. All the discussions above are confirmed by six different standard models in the literature.

- Besides, of the theoretical approaches, the indentation-induced cracking model with the closest findings exhibits the highest performance to scrutinize the load-independent Vickers microindentation hardness parameters in the plateau limit regions. Finally, the obtained scientific information deduced from this work verifies the importance and power of theoretical modeling approaches on the mechanical performance and characterization as well as the serious advantages such as the free, simple, fast, practical and reliable of models.

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