Synthesis, microstructure and indentation Vickers hardness for $(Y_3Fe_5O_{12})_x/Cu_{0.5}Ti_{0.5}Ba_2Ca_2Cu_3O_{10-\delta}$ composites

M. Rekaby¹ · N. H. Mohammed¹ · M. Ahmed² · A. I. Abou-Aly¹

Received: 10 November 2021 / Accepted: 12 February 2022 / Published online: 6 March 2022
© The Author(s) 2022

Abstract

High-performance and low-cost composites are engineers' dream for technological applications. To fulfill the material for an engineering application, it is important to understand the mechanical properties of the material. The primary goal of this research is to investigate the impact of nano-sized Yttrium iron garnet ($Y_3Fe_5O_{12}$) addition on the microstructure and mechanical properties of the polycrystalline $Cu_{0.5}Ti_{0.5}Ba_2Ca_2Cu_3O_{10-\delta}$ (CuTi-1223) superconductor. Co-precipitation and solid-state reaction methods were utilized to prepare $Y_3Fe_5O_{12}$ nanoparticles and $Cu_{0.5}Ti_{0.5}Ba_2Ca_2Cu_3O_{10-\delta}$ superconductor, respectively. $(Y_3Fe_5O_{12})_x/Cu_{0.5}Ti_{0.5}Ba_2Ca_2Cu_3O_{10-\delta}$ nanoparticle/superconductor composites were formed by adding small contents of $Y_3Fe_5O_{12}$ ($x=0.00, 0.02, 0.04, 0.06, 0.08$, and 0.10 wt%) to the CuTi-1223 matrix. The volume fraction percentage of the main phase, CuTi-1223, was increased from 87.9 to 91.4% as x was adjusted from 0.00 to 0.04 wt%. The unit cell parameters (**a** and **c**) remained unchanged following the addition of $Y_3Fe_5O_{12}$ nanoparticles to the host CuTi-1223. The porosity percentage ($P\%$) was decreased from 39.1 to 29.4% as x was increased from 0.00 to 0.10 wt%. Thus, the addition of $Y_3Fe_5O_{12}$ nanoparticles has the ability to reduce weak links and voids among the CuTi-1223 superconducting grains. The grain morphology for the prepared composites was identified through scanning electron microscopy. The different elemental compositions were detected by energy-dispersive X-ray measurements. Vickers microindentation hardness test was employed to study the mechanical strength of the prepared composites. Analysis and modelling of Vickers hardness (H_v) versus test load (F) were done through various models. Meyer's empirical law showed that all the prepared composites follow normal indentation size effect behaviour. Hays and Kendall model clarified that the applied test load was sufficient to produce both elastic and plastic deformation for the investigated samples. The elastic/plastic deformation model indicated that the prepared samples contain an elastic relaxation portion that recovers after withdrawing the test load. The proportional sample resistance and modified proportional sample resistance models confirmed the HK model findings. Moreover, the HK model was found to be the most suitable model for describing the microhardness results of the prepared samples. Furthermore, the elastic modulus (E), yield strength (Y), fracture toughness (K) and brittleness index (B) for the prepared composites were calculated as function of $Y_3Fe_5O_{12}$ addition.

Keywords Yttrium iron garnet · CuTi-1223 · Vickers hardness · Elastic modulus · Yield strength

1 Introduction

A high superconducting transition temperature, T_c , is usually the first sign of progress in the quest for a new superconductor. However, all superconductor power applications, as well as the majority of other applications, necessitate high current

carrying capacity, as measured by the critical current density J_c ($>10^5$ A/cm²), in the presence of significant magnetic fields (10–20 T) at 77 K, as defined by the critical magnetic field H_c [1]. The three critical parameters (T_c , J_c and H_c) mentioned so far are certainly not the only important ones for practical applications. Mechanical properties are also critical, since large magnetic fields and machine motions (e.g., centripetal forces) may produce substantial forces on the superconductor which must be well accounted for if damage is to be avoided. Cyclic forces may produce fatigue of the superconductor. Ductility, malleability, hardness and strain tolerance affect the ease with which the wires may be manufactured and then wound into desired coil shapes.

✉ M. Rekaby
mona2sci@yahoo.com

¹ Department of Physics, Faculty of Science, Alexandria University, Alexandria, Egypt

² Department of Basic Sciences, Faculty of Engineering, Pharos University, Alexandria, Egypt

Superconductor-based products, in comparison to their traditional counterparts, are particularly environmentally friendly. They are also often at least 50% smaller and lighter than comparable conventional units, which translate to cost savings. High-temperature superconductors (HTS) have a wide range of very sensitive and reliable advanced technological applications such as in vivo live body measurements in medicine, terahertz equipment for security systems, quantum bit applications in quantum computers and bolometers for some space research. HTS also have been reliably utilized in magnetic resonance imaging (MRI), magnetic resonance spectroscopy (MRS), magnetoencephalography (MEG) and magnetocardiography (MCG) for both analysis of magnetic activity of different regions of the human body such as brain and heart wave activities and very early diagnosis of several diseases [2]. Furthermore, one of the most important usages of HTS materials is the use of superconducting cables in high voltage transmission lines. The quality of HTS tapes determines the performance of HTS cable. HTS tapes for power transmission cables must be manufactured in a sufficient length to accommodate the requisite length of cable core to be placed.

The $\text{Cu}_{0.5}\text{Tl}_{0.5}\text{Ba}_2\text{Ca}_2\text{Cu}_3\text{O}_{10-\delta}$, CuTl-1223, phase is a Cu and Tl mixed-based superconductor [1]. It is the most appealing phase in its family and a potential candidate from an application standpoint due to its relatively large critical current density J_c ($> 10^5 \text{ A/cm}^2$), high superconducting transition temperature T_c ($T_c \sim 106\text{--}132 \text{ K}$) close to that of Hg-1223 [2], low superconducting anisotropy ($\gamma = \xi_{ab}/\xi_c$), long coherence length along c -axis (ξ_c) and small penetration depth (λ) [3]. Besides all the superior characteristics mentioned for CuTl-1223 phase, CuTl-1223 must possess appropriate mechanical strength that gives it the ability to be flexible and moldable for cables, coils and tapes usage.

The low-cost synthesis of CuTl-1223 at ambient pressure is a suitable method for large-scale manufacturing. The main drawback of this synthesis process is the existence of voids and holes in increased density, which affects total superconducting critical parameters. This problem is most successfully treated by enhancing grain connection and decreasing the density of voids and pores by the incorporation of different nanoparticles, which is the simplest and most efficient method in this regard [2, 3]. Nanoparticle addition has previously been proven by many scientists to be useful tool for improving the microstructure and mechanical characteristics of CuTl-1223 superconducting phase [4]. Mohammed et al. [5] conducted a comparative investigation of the effects of nano- SnO_2 and nano- In_2O_3 addition on CuTl-1223. The results indicated that adding nano- SnO_2 up to 1.0 wt% improved the mechanical characteristics of CuTl-1223, whereas just a little amount of nano- In_2O_3 , $x=0.1 \text{ wt\%}$, increased the phase's Vickers hardness. The impact of nano- BaSnO_3 addition in CuTl-1223 superconducting bulk was examined by Srouf et al. [6]. The results indicated that microindentation Vickers hardness values rose up to $x=0.25 \text{ wt\%}$, then dropped with a

further rise in x till 1.0 wt%. Awad [7] investigated the effect of nano-MgO addition on the electrical and mechanical properties of the CuTl-1223 phase and it was concluded that Vickers hardness increased with increasing MgO content up to 0.6 wt%, but then decreased as MgO content increased further.

Microindentation Vickers hardness is a non-destructive test that determines a material's ability to resist plastic deformation by indentation. The Vickers indentation method [8–13] is the most widely used microindentation method. In this method, a square-based pyramidal diamond indenter is used on the sample's surface under regulated stress and time rate conditions. After the load is removed, the average diagonal length (d) of the resultant imprint is measured and linked to the microhardness number (H_v).

In this study, the influence of nano-sized $\text{Y}_3\text{Fe}_5\text{O}_{12}$ addition on the microstructure, morphology, elemental compositions and mechanical properties of $\text{Cu}_{0.5}\text{Tl}_{0.5}\text{Ba}_2\text{Ca}_2\text{Cu}_3\text{O}_{10-\delta}$ superconductor was studied. We have examined the ability of $\text{Y}_3\text{Fe}_5\text{O}_{12}$ to improve the intergrain connectivity and in turn the mechanical properties of the host superconducting phase. This can be done by healing the voids among superconductor grains with these nanoparticles. The microindentation Vickers hardness test estimated numerous important mechanical parameters such as Vickers hardness (H_v), elastic modulus (E), yield strength (Y), fracture toughness (K) and brittleness index (B) for $(\text{Y}_3\text{Fe}_5\text{O}_{12})_x/\text{Cu}_{0.5}\text{Tl}_{0.5}\text{Ba}_2\text{Ca}_2\text{Cu}_3\text{O}_{10-\delta}$ composites, with $x=0.00, 0.02, 0.04, 0.06, 0.08$ and $0.10 \text{ wt\%}$. Furthermore, the indentation size effect (ISE) behaviour was analysed using various models.

2 Experimental techniques

2.1 Nano- $\text{Y}_3\text{Fe}_5\text{O}_{12}$ preparation

The co-precipitation technique was used to produce nano-sized $\text{Y}_3\text{Fe}_5\text{O}_{12}$ from the starting components $\text{YCl}_3 \cdot 6\text{H}_2\text{O}$ and $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$. The powder was bought from Sigma-Aldrich and was of excellent purity. The powders were dissolved in 100 ml of distilled water in the stoichiometric ratio of Y: Fe 3:5. With magnetic stirring, 5 M NaOH was added to the solution dropwise until the pH reached 12. The solution is then heated for 4 h at 80°C while being constantly stirred. After that, the samples were rinsed until the pH reached 7, as determined by a digital pH metre. The samples were then dried at 100°C for 18 h. Finally, these samples were calcined for 2 h at 1050°C . A schematic diagram for $\text{Y}_3\text{Fe}_5\text{O}_{12}$ preparation is shown in Fig. 1a.

2.2 $(\text{Y}_3\text{Fe}_5\text{O}_{12})_x/\text{CuTl-1223}$ preparation

A solid-state reaction technique was used to synthesize $\text{Cu}_{0.5}\text{Tl}_{0.5}\text{Ba}_2\text{Ca}_2\text{Cu}_3\text{O}_{10-\delta}$ superconducting samples at ambient pressure. High-quality starting materials of Tl_2O_3 , BaO_2 , CaO , and CuO were crushed and filtered through a $65 \mu\text{m}$

Fig. 1 Schematic diagram for **a** Y₃Fe₅O₁₂ co-precipitation method, **b** (Y₃Fe₅O₁₂)_x/Cu_{0.5}Ti_{0.5}Ba₂Ca₂Cu₃O_{10-δ} composites preparation steps

sieve in their nominal compositions. To make (Y₃Fe₅O₁₂)_x/Cu_{0.5}Ti_{0.5}Ba₂Ca₂Cu₃O_{10-δ} composites, Y₃Fe₅O₁₂ nanoparticles (~50 to 80 nm) with weight per cent 0.00, 0.02, 0.04, 0.06, 0.08, and 0.10 wt% were mixed and crushed to ensure homogeneity. The powder was compacted into a disc and then covered in a silver sheet to decrease Ti losses during the heating operation. The samples were sintered in a sealed quartz tube at a rate of 2 °C/min to 760 °C, then at a rate of 1 °C/min to 850 °C for 6 h. The samples were gently cooled to room temperature at the end of this stage. The preparation steps are summarized in Fig. 1b.

2.3 characterization and measurements

Shimadzu-7000 XRD with CuKα radiation ($\lambda = 1.5418 \text{ \AA}$), scan range of 10°–80°, step size of 0.04° and scan rate of 4°/min in a continuous scan mode was used to identify (Y₃Fe₅O₁₂)_x/Cu_{0.5}Ti_{0.5}Ba₂Ca₂Cu₃O_{10-δ} composites. JEOL scanning electron microscope model JSM-IT200 was used

to identify the material's morphology and elemental content. The device operated at high voltage 20–30 kV, at which low pressure was attained using a rotary pump. The electron source was at a distance of 15 mm from the surface of the sample. The value of magnification at which the pictures were taken is 1000. A digital microhardness tester model IN-412A was used to determine Vickers microhardness. Vickers indenter has a square-pyramid diamond shape. The indenter was used on the sample surface with test loads ranging from 0.49 to 2.94 N and a dwell period of 30 s. For each individual load, the average value of 5 indentations at different points on the sample surface was obtained to ensure accuracy of microhardness data. Vickers indentation marks were roughly square in form with two diagonals remained on the sample surface after unloading. After unloading, the length of the two diagonals was estimated using a calibrated micrometre eyepiece fitted on the microscope. With an accuracy of $\pm 0.1 \text{ }\mu\text{m}$, the diagonal lengths of indentation were calculated and were connected to an average value of Vickers hardness number (H_v).

3 Results and discussion

3.1 XRD

Figure 2 shows XRD patterns for $(Y_3Fe_5O_{12})_x/Cu_{0.5}Ti_{0.5}Ba_2Ca_2Cu_3O_{10-\delta}$ with $x=0.00, 0.02, 0.04, 0.06, 0.08$, and 0.10 wt%. The majority of the diffraction peaks are clearly indexed by the tetragonal indices of CuTi-1223 phase with P4/mmm symmetry. There are no peaks for Yttrium or iron-based compounds that were detected in the patterns. This shows that the structure of the CuTi-1223 phase is not affected by the $Y_3Fe_5O_{12}$ nanoparticles. The relative volume fraction percentages for CuTi-1223, CuTi-1212 and $BaCuO_2$ phases are determined as follows:

$$\begin{aligned} \text{CuTi-1223\%} &= \frac{\Sigma I(\text{CuTi-1223})}{\Sigma I(\text{CuTi-1223}) + \Sigma I(\text{CuTi-1212}) + \Sigma I(\text{BaCuO}_2)} \times 100, \\ \text{CuTi-1212\%} &= \frac{\Sigma I(\text{CuTi-1212})}{\Sigma I(\text{CuTi-1223}) + \Sigma I(\text{CuTi-1212}) + \Sigma I(\text{BaCuO}_2)} \times 100 \\ \text{BaCuO}_2\% &= \frac{\Sigma I(\text{BaCuO}_2)}{\Sigma I(\text{CuTi-1223}) + \Sigma I(\text{CuTi-1212}) + \Sigma I(\text{BaCuO}_2)} \times 100, \end{aligned} \quad (1)$$

where ΣI is the sum of the intensities of the diffraction peaks [14]. The relative volume fraction percentages for $(Y_3Fe_5O_{12})_x/Cu_{0.5}Ti_{0.5}Ba_2Ca_2Cu_3O_{10-\delta}$ with $x=0.00, 0.02, 0.04, 0.06, 0.08$, and 0.10 wt% are shown in Table 1. The volume fraction percentage of CuTi-1223 phase increases from 87.9 to 91.4% as $Y_3Fe_5O_{12}$ nanoparticles addition (x) is adjusted from 0.00 to 0.04 wt%. Higher $Y_3Fe_5O_{12}$ addition contents, $x > 0.04$ wt%, cause a delay in the phase percentage of CuTi-1223 and enhance CuTi-1212 and $BaCuO_2$ phase percentages. Similar findings were obtained when Al_2O_3 and MgO nanoparticles were added to the Bi-2223 phase [15, 16]. They ascribe the drop in volume percentage for higher nanoparticle additions to solid nanoparticles ability to inhibit spatial phase development to some limit. This delay

Fig. 2 XRD patterns for $(Y_3Fe_5O_{12})_x/Cu_{0.5}Ti_{0.5}Ba_2Ca_2Cu_3O_{10-\delta}$ with $x=0.00, 0.02, 0.04, 0.06, 0.08$ and 0.10 wt%

Table 1 Variation of Volume fraction % and unit cell parameters of $(Y_3Fe_5O_{12})_x/Cu_{0.5}Ti_{0.5}Ba_2Ca_2Cu_3O_{10-\delta}$ composites with $x=0.00, 0.02, 0.04, 0.06, 0.08$ and 0.10 wt. %

x (wt%)	CuTi-1223 (%)	CuTi-1212 (%)	BaCuO ₂ (%)	a (Å)	c (Å)
0.00	87.9	3.02	8.20	3.8515	15.878
0.02	88.3	2.04	8.99	3.8514	15.879
0.04	91.4	1.18	7.14	3.8517	15.868
0.06	87.6	2.10	9.99	3.8511	15.867
0.08	86.4	3.51	9.89	3.8517	15.876
0.10	83.2	4.89	11.8	3.8513	15.879

Fig. 3 Variation of porosity with x for $(Y_3Fe_5O_{12})_x/Cu_{0.5}Ti_{0.5}Ba_2Ca_2Cu_3O_{10-\delta}$ composites

might also be attributed to the ferrimagnetic character of $Y_3Fe_5O_{12}$ nanoparticles, which promotes Cooper pair breaking and encourages the retardation of CuTi-1223 phase formation at high concentrations. Table 1 displays the estimated values of the unit cell parameters (a and c). The unit cell parameters remained unchanged following the addition of $Y_3Fe_5O_{12}$ nanoparticles to the host CuTi-1223 material. This also shows that $Y_3Fe_5O_{12}$ preferentially inhabit the interstitial areas between the grains. Kong et al. [17] and Albiss et al. [18] found similar findings when adding $NiFe_2O_4$ and NiO nanoparticles to the $Bi_{1.6}Pb_{0.4}Sr_2Ca_2Cu_3O_{10+\delta}$ phase.

The porosity percentage (P %) of the synthesized composites is theoretically determined using the following equation:

$$P(\%) = \left(1 - \frac{D_e}{D_T}\right) \times 100, \quad (2)$$

where D_e and D_T are the experimental and theoretical densities, respectively.

The variation of porosity with x for $(Y_3Fe_5O_{12})_x/Cu_{0.5}Ti_{0.5}Ba_2Ca_2Cu_3O_{10-\delta}$ is shown in Fig. 3. It is clear that as x increases from 0.00 to 0.10 wt.%, P decreases from 39.1 to 29.4%. Thus, the addition of $Y_3Fe_5O_{12}$ nanoparticles has the ability to reduce weak links and voids among the CuTi-1223 superconducting grains. The enhancement in the nano-sized $Y_3Fe_5O_{12}$ content and its uniform distribution will assist its penetration ability within the pores and voids of the hybrid composite matrix leading to a linear increase in the experimental density and a gradual decrease in porosity. Khalil [19] reported that for $Bi_{1.6}Pb_{0.4}Sr_2Ca_{2-x}Sm_xCu_3O_y$, the decrease in porosity may be related to an increase in grain coupling strength and degree of grain orientation of the samples. Awad [7] investigated similar porosity results for CuTi-1234 added with MgO nanoparticles.

3.2 SEM and EDX

SEM micrographs of $(Y_3Fe_5O_{12})_x/Cu_{0.5}Ti_{0.5}Ba_2Ca_2Cu_3O_{10-\delta}$ composites with $x=0.00, 0.02, 0.04, 0.06, 0.08$ and 0.10 wt% were used to examine the morphology, as shown in Fig. 4a–f. The CuTi-1223 phase formation was confirmed from the observed plate-like grains. Some irregular shaped grains were assigned for impurity phases such as CuTi-1212 and BaCuO₂. Furthermore, even after high-temperature sintering, there are fine spherical grains that may indicate the presence of $Y_3Fe_5O_{12}$ nanoparticles in CuTi-1223 matrix. Also, the micrographs showed apparent suppression in the intergrain voids and improvement in the intergrain weak links that can be visualized in comparison with the pure sample (Fig. 4a). Solid-state diffusional phase transformation, which includes nucleation, growth, and impingement, is directly responsible for the arrangements of constituent composites, which include a specific volume fraction of spatially distributed phases with various compositions, grain size and morphology with random orientation, and their spatial distribution [20]. Numerical simulation approaches may be employed, in future scope of the current work, to account for experimental condition limitations and dynamically recreate microstructure development. Empirical and analytical models [20], phase-field models [21, 22], cellular automaton models, Monte Carlo models, and molecular dynamics approaches [20] are now the most sophisticated numerical simulation methods. In general, empirical and analytical models provide more average information, such as average grain size and phase volume fraction of microstructure development at the macroscale. Mesoscopic models, such as phase-field models, cellular automata, and Monte Carlo models, on the other hand, can recreate the development of grain shape and spatial distribution, as well as the solute concentration field, in addition to providing macroscopic

Fig. 4 SEM micrographs for $(Y_3Fe_5O_{12})_x/Cu_{0.5}Tl_{0.5}Ba_2Ca_2Cu_3O_{10-\delta}$ **a** $x=0.00$, **b** $x=0.02$, **c** $x=0.04$, **d** $x=0.06$ wt%, **e** $x=0.08$ and **f** $x=0.10$ wt%

Fig. 5 EDX spectra for $(Y_3Fe_5O_{12})_x/Cu_{0.5}Tl_{0.5}Ba_2Ca_2Cu_3O_{10-\delta}$ **a** $x=0.00$ and **b** $x=0.10$ wt%

average information [20]. The phase-field approach has grown in importance and versatility for modelling solidification, precipitate development, coarsening, grain growth, martensitic transformations, dislocation dynamics, crack propagation, and other phenomena.

Figure 5a and b shows the EDX analysis of $(Y_3Fe_5O_{12})_x/Cu_{0.5}Tl_{0.5}Ba_2Ca_2Cu_3O_{10-\delta}$ composites with $x = 0.00$ and 0.10 wt%, respectively. It should be noted that no peaks corresponding to Yttrium or iron-based compounds were found in the XRD patterns (as seen from Fig. 2). However, EDX analysis shows the minor elemental content of Yttrium and iron elements in the bulk sample, as shown in Fig. 5b. This means that they do not enter the structure of the $CuTl-1223$ phase, but rather occupy the interstitial space between the grains. The nearly unchangeable unit cell parameters support this conclusion. Guilmeau et al. [23] discovered similar results when they investigated the effect of MgO nanoparticles addition into the Bi-2223 superconducting phase. The elemental contents of different elements present in $(Y_3Fe_5O_{12})_x/Cu_{0.5}Tl_{0.5}Ba_2Ca_2Cu_3O_{10-\delta}$; $x = 0.00$ and $x = 0.10$ wt% composites are given in Table 2.

3.3 Microindentation Vickers hardness

Vickers hardness (H_v) for $(Y_3Fe_5O_{12})_x/Cu_{0.5}Tl_{0.5}Ba_2Ca_2Cu_3O_{10-\delta}$, with $x = 0.00, 0.02, 0.04, 0.06, 0.08$, and 0.10 wt%, is computed from the following formula [24]:

$$H_v = 1854.4 \left(\frac{F}{d^2} \right) \text{ (GPa)}, \quad (3)$$

where F is the applied force in Newton and d is the average diagonal length of indentation in micrometres. Figure 6 depicts the change of room temperature Vickers hardness (H_v) with test load (F), at dwell time $t = 30$ s for $(Y_3Fe_5O_{12})_x/Cu_{0.5}Tl_{0.5}Ba_2Ca_2Cu_3O_{10-\delta}$, with $x = 0.00, 0.02, 0.04, 0.06, 0.08$, and 0.10 wt%. The variation could be divided into two segments: a load dependent segment that emerges at

Table 2 The elemental contents of different elements present in $(Y_3Fe_5O_{12})_x/Cu_{0.5}Tl_{0.5}Ba_2Ca_2Cu_3O_{10-\delta}$; $x = 0.00$ and $x = 0.10$ wt% composites

Element	Mass % for $x = 0.00$ wt%	Mass % for $x = 0.10$ wt%
O	17.89 ± 0.23	18.28 ± 0.43
Ca	8.17 ± 0.14	8.93 ± 0.27
Cu	28.14 ± 0.44	29.28 ± 0.70
Ba	31.31 ± 0.38	28.93 ± 0.41
Tl	14.48 ± 0.27	13.94 ± 0.41
Y	—	0.62 ± 0.20
Fe	—	0.01 ± 0.06
Total (%)	100	100

low applied loads $F < 1$ N and declines fast as the applied load increases, and a load independent segment (nearly plateau) that appears at higher values $F > 1$ N. This variation is known as indentation size effect (ISE). Moreover, H_v is high at low indentation loads, which can be attributed to the material weak grain boundaries [25]. Furthermore, it was found, for all the applied loads (F), that H_v increases with increasing the $Y_3Fe_5O_{12}$ content up to $x = 0.10$ wt% which may be attributed to the ability of these nanoparticles to heal microcracks and voids among superconducting grains. This healing in microstructure was confirmed from SEM results.

3.4 Analysis and Modelling

3.4.1 Meyer's law

Meyer's law [26, 27] might explain the nonlinear load dependent segment, which offers a relationship between the applied force (F) and the indentation diagonal length (d) as:

$$F = Ad^n, \quad (4)$$

Fig. 6 Vickers hardness (H_v) versus test load (F) for $(Y_3Fe_5O_{12})_x/Cu_{0.5}Tl_{0.5}Ba_2Ca_2Cu_3O_{10-\delta}$, with $x = 0.00, 0.02, 0.04, 0.06, 0.08$, and 0.10 wt%

Fig. 7 Meyer's law analysis for $(Y_3Fe_5O_{12})_x/Cu_{0.5}Tl_{0.5}Ba_2Ca_2Cu_3O_{10-\delta}$, with $x = 0.00, 0.02, 0.04, 0.06, 0.08$, and 0.10 wt%

Table 3 Fitting parameters obtained from Meyer, HK, EPD, PSR and MPSR with x

Model	Meyer		HK		EPD		PSR		MPSR		
	n	$A \times 10^{-3}$ (GPa)	W (N)	$A_1 \times 10^{-4}$ (N/(μm) ²)	$A_2 \times 10^{-5}$ (N/(μm) ²)	d_0 (μm)	$\alpha \times 10^{-2}$ (N/ μm)	$\beta \times 10^{-5}$ (N/(μm) ²)	α_2 (N)	α_3 (N/ μm)	$\alpha_4 \times 10^{-4}$ (N/(μm) ²)
0.00	1.10	11.79	0.39	1	8.1	46	1.54	4	0.50	0.0037	2
0.02	1.23	9.08	0.35	3	17.16	27.5	1.6	10	0.420	0.0033	3
0.04	1.33	7.78	0.32	4	27.55	19.83	1.69	20	0.312	0.0055	4
0.06	1.15	13.9	0.42	3	16.9	32.54	2.02	9	0.264	0.0070	2
0.08	1.48	5.94	0.30	6	48.4	12.68	1.7	40	0.003	0.017	4
0.10	1.57	5.02	0.25	9	72.9	8.47	1.59	70	0.020	0.0146	7

Fig. 8 Hardness data for $(\text{Y}_3\text{Fe}_5\text{O}_{12})_x/\text{Cu}_{0.5}\text{Ti}_{0.5}\text{Ba}_2\text{Ca}_2\text{Cu}_3\text{O}_{10-\delta}$, with $x=0.00, 0.02, 0.04, 0.06, 0.08$, and 0.10 wt%, fitted according to HK model **Fig. 9** Hardness data for $(\text{Y}_3\text{Fe}_5\text{O}_{12})_x/\text{Cu}_{0.5}\text{Ti}_{0.5}\text{Ba}_2\text{Ca}_2\text{Cu}_3\text{O}_{10-\delta}$, with $x=0.00, 0.02, 0.04, 0.06, 0.08$, and 0.10 wt%, fitted according to EPD model

where n is Meyer index and A is the hardness constant. Meyer index (n) is commonly employed as an indication of the ISE trend: when $n < 2$, microhardness decreases with applied load (normal ISE); when $n > 2$, microhardness increases with applied load (reverse ISE); and when $n = 2$, microhardness is independent of applied stress. Plots of $\ln F$ versus $\ln d$ for $(\text{Y}_3\text{Fe}_5\text{O}_{12})_x/\text{Cu}_{0.5}\text{Ti}_{0.5}\text{Ba}_2\text{Ca}_2\text{Cu}_3\text{O}_{10-\delta}$, with $x=0.00, 0.02, 0.04, 0.06, 0.08$, and 0.10 wt%, is shown in Fig. 7. The n -values for all samples were found to be less than 2 (see Table 3), confirming that the investigated samples exhibit normal ISE (NISE) behaviour.

3.4.2 Hays and Kendall model

Hays and Kendall (HK) model [28] claims that only plastic deformation is visible at H_v levels above a threshold value W (sample resistance pressure). According to Gane and Bowden [29], the indenter does not penetrate the sample surface until it reaches a threshold value of applied load. Instead of the applied load, the effective load was connected to indentation size in this model by $F_{\text{eff}} = F - W$, and therefore, the load dependency of indentation size was provided by

$$F = W + A_1 d^2 \quad (5)$$

where A_1 is a constant that is unaffected by the applied load.

Figure 8 displays hardness data for $(\text{Y}_3\text{Fe}_5\text{O}_{12})_x/\text{Cu}_{0.5}\text{Ti}_{0.5}\text{Ba}_2\text{Ca}_2\text{Cu}_3\text{O}_{10-\delta}$, with $x=0.00, 0.02, 0.04, 0.06, 0.08$, and 0.10 wt%, fitted according to HK model. The data show an excellent linear relationship with very high regression coefficients 99.9%. The values of A_1 and W are tabulated in Table 3. The W values reported for all samples were positive, demonstrating the production of elastic and plastic deformation with a sufficient applied force [30]. This suggests that for the prepared composites, the minimum test load required to induce plastic deformation is higher than the estimated values ($W=0.39, 0.35, 0.32, 0.42, 0.30$, and 0.25 N). The results are consistent with the range of test loads used, which vary from 0.49 to 2.94 N. As a result, W values are even lower than the first low test load ($F=0.49$ N). Furthermore, with the addition of $\text{Y}_3\text{Fe}_5\text{O}_{12}$ nanoparticles, the values of A_1 followed the same pattern as the fluctuation of H_v with F , confirming the NISE trend.

3.4.3 Elastic/plastic deformation model

Elastic/plastic deformation (EPD) model stated that the indentation comprises an elastic part that occurs after the indenter is withdrawn at the nearness indentation imprint [31]. As a result, an elastic component d_0 is added to the measured plastic component (d) as follows:

$$F = A_2(d + d_0)^2, \quad (6)$$

where A_2 is constant. Figure 9 depicts the change of $F^{0.5}$ versus d plots for $(Y_3Fe_5O_{12})_x/Cu_{0.5}Ti_{0.5}Ba_2Ca_2Cu_3O_{10-\delta}$, with $x=0.00, 0.02, 0.04, 0.06, 0.08$, and 0.10 wt%. It is clear that the data are well fitted to Eq. (6) with linear regression coefficients greater than 99.9%. Table 3 shows the estimated values of A_2 and d_0 based on the fitting of experimental findings according to Eq. (6). As a result, the value of d_0 was positive for all samples, indicating that for this range of applied loads, elastic deformation was seen in addition to plastic deformation, indicating that elastic relaxation was present among the prepared samples [32]. Their values, however, are far too large to be accepted as a correction for elastic recovery in the measured plastic indentation. As a result, it can be concluded that the indentation size error caused by the blunting of the indenter tip and the elastic recovery after indentation are not the only causes of the ISE behaviour observed in the prepared composites.

3.4.4 Proportional sample resistance model

The proportional sample resistance (PSR) model is a modification of the HK model in which the term αd replaces the load independent constant W . This model was used by Li and Bradt [33] to describe the ISE behaviour of various

materials. The indentation load dependent and independent Vickers hardness values of the PSR model are provided by

$$F = \alpha d + \beta d^2. \quad (7)$$

The constant α is composed of two components: the elastic resistance of the test specimen and the friction resistance generated at the indenter facet/specimen contact [33]. β is a plastic property constant that is used to compute the theoretical value of microhardness. Figure 10 depicts a plot of (F/d) versus d for $(Y_3Fe_5O_{12})_x/Cu_{0.5}Ti_{0.5}Ba_2Ca_2Cu_3O_{10-\delta}$, with $x=0.00, 0.02, 0.04, 0.06, 0.08$, and 0.10 wt%. The values of α and β are determined using Eq. (7), where β represents the slope and α represents the y-intercept, and their values are listed in Table 3. The values of α and β displayed unsystematic fluctuation with $Y_3Fe_5O_{12}$ contents. The tabulated data revealed that the values of α for all samples were positive, validating the HK method results. These findings indicate that elastic deformation occurred in $(Y_3Fe_5O_{12})_x/Cu_{0.5}Ti_{0.5}Ba_2Ca_2Cu_3O_{10-\delta}$ composites in addition to plastic deformation. Furthermore, α increased when x rose up to 0.06 wt%, which was attributable to energy dissipation through fractures at the interfaces [24].

3.4.5 Modified proportional sample resistance model

Taking the influence of the machining induced plastically deformed surface on microhardness measurements into account, it was recommended [34] that the PSR model be modified into the following equation:

$$F = \alpha_2 + \alpha_3 d + \alpha_4 d^2 \quad (8)$$

where α_2 is the lowest applied stress causing indentation in the samples. It relates to the HK model's threshold load constant W and is linked to surface residual stresses caused by surface machining and polishing. The physical meaning

Fig. 10 Hardness data for $(Y_3Fe_5O_{12})_x/Cu_{0.5}Ti_{0.5}Ba_2Ca_2Cu_3O_{10-\delta}$, with $x=0.00, 0.02, 0.04, 0.06, 0.08$, and 0.10 wt%, fitted according to PSR model

Fig. 11 Hardness data for $(Y_3Fe_5O_{12})_x/Cu_{0.5}Ti_{0.5}Ba_2Ca_2Cu_3O_{10-\delta}$, with $x=0.00, 0.02, 0.04, 0.06, 0.08$, and 0.10 wt%, fitted according to MPSR model

Fig. 12 Experimental plateau Hardness data with the estimated true hardness from HK, EPD, PSR, MPSR models for $(Y_3Fe_5O_{12})_x/Cu_{0.5}Ti_{0.5}Ba_2Ca_2Cu_3O_{10-\delta}$, with $x=0.00, 0.02, 0.04, 0.06, 0.08$, and 0.10 wt%, fitted according to PSR model

Table 4 Mechanical parameters E , Y , K , and B for all samples $(Y_3Fe_5O_{12})_x/Cu_{0.5}Ti_{0.5}Ba_2Ca_2Cu_3O_{10-\delta}$ with $x=0.00, 0.02, 0.04, 0.06, 0.08$, and 0.10 wt%

E (GPa)	Y (GPa)	K (GPa $\mu m^{0.5}$)	B ($\mu m^{-0.5}$)
25.01	0.10	0.09	3.14
43.57	0.17	0.13	4.07
47.27	0.19	0.13	4.13
67.27	0.27	0.18	4.50
107.55	0.43	0.21	6.21
148.53	0.60	0.24	7.54

of parameters α_3 and α_4 is the same as it is in the PSR model [35].

Figure 11 depicts the change of F with d for $(Y_3Fe_5O_{12})_x/Cu_{0.5}Ti_{0.5}Ba_2Ca_2Cu_3O_{10-\delta}$, with $x=0.00, 0.02, 0.04, 0.06, 0.08$, and 0.10 wt%. Table 3 contains the MPSR parameters α_2 , α_3 and α_4 , which were derived using a standard polynomial fit to the data. The positive values of α_2 clarify that the samples suffer from both elastic and plastic deformation [36].

The true microhardness or the microhardness in plateau region can be estimated from the previously stated models, i.e. HK, EPD, PSR and MPSR, [32] according to the following relations

$$H_{HK} = 1854.4 \times A_1 \quad (9)$$

$$H_{EPD} = 1854.4 \times A_2 \quad (10)$$

$$H_{PSR} = 1854.4 \times \beta \quad (11)$$

$$H_{MPSR} = 1854.4 \times \alpha_4, \quad (12)$$

A comparison between the experimental H_v values with the theoretically estimated H_v values, estimated from HK, EPD,

PSR, MPSR models according to Eqs. 9–12, is shown in Fig. 12 for $(Y_3Fe_5O_{12})_x/Cu_{0.5}Ti_{0.5}Ba_2Ca_2Cu_3O_{10-\delta}$, with $x=0.00, 0.02, 0.04, 0.06, 0.08$, and 0.10 wt%. It is clear, from Fig. 12, that HK model gives very close results to our experimental H_v values so it is the best suitable model for describing true hardness data of $(Y_3Fe_5O_{12})_x/Cu_{0.5}Ti_{0.5}Ba_2Ca_2Cu_3O_{10-\delta}$ composites.

The elastic modulus (E), yield strength (Y), fracture toughness (K) and brittleness index (B) were shown to be linked to true microhardness (H_v) as $E = 81.96 \times H_v$, $Y = \frac{H_v}{3}$, $K = \sqrt{2E\alpha}$ and $B = \frac{H_v}{K}$, respectively [37]. Table 4 shows the estimated values of the mechanical parameters E , Y , K , and B for $(Y_3Fe_5O_{12})_x/Cu_{0.5}Ti_{0.5}Ba_2Ca_2Cu_3O_{10-\delta}$, with $x=0.00, 0.02, 0.04, 0.06, 0.08$, and 0.10 wt%. Table 4 shows that E , Y , K , and B follow the same trend as the change of H_v with x . Zewnet et al. [38] examined the elastic modulus-critical grain size relationship to identify the critical grain size at which microcracking occurs owing to anisotropic thermal stresses that emerge during processing. The stronger the connection between atoms or molecules in materials, the greater the value of E and hence Y of a sample. The fracture toughness K of a material is defined as its capacity to withstand fractures. The rise in K is proportional to the increase in surface energy. The brittleness index, which measures the ability of a material to break when subjected to deformation, is also an essential practical parameter.

4 Conclusions

Series of $(Y_3Fe_5O_{12})_x/CuTi-1223$ composites with $x=0.00, 0.02, 0.04, 0.06, 0.08$ and 0.10 wt% were successfully synthesized by solid-state reaction to confirm their reproducibility. The crystal structure of the host $CuTi-1223$ superconducting matrix was not affected with the addition of $Y_3Fe_5O_{12}$ nanoparticles. The impact of $Y_3Fe_5O_{12}$ addition on the mechanical characteristics of $CuTi-1223$ composites such as Vickers hardness, elastic modulus, brittleness index, yield strength, and fracture toughness has been studied. The results demonstrate that $Y_3Fe_5O_{12}$ addition improves the mechanical characteristics of $CuTi-1223$ composites. Meyer's law, HK, EPD, PSR, and MPSR models were used to assess the experimental results of the microhardness measurements. HK model was found to be the most suitable model describing the hardness data of the prepared samples. As the amount of $Y_3Fe_5O_{12}$ in the samples was increased, the elastic modulus (E), yield strength (Y), fracture toughness (K) and brittleness index (B) of the samples were improved. These enhancements are thought to be due to the presence of $Y_3Fe_5O_{12}$ particles, which can encourage compressive stresses in the superconducting matrix and resist crack propagation by pinning the propagating cracks.

Acknowledgements Special thanks to the spiritual father of the work, Professor R. Awad for his kind assistance in $Y_3Fe_5O_{12}$ preparation. This work was performed in *Superconductivity and metallic glasses laboratory*, Faculty of Science, Alexandria University, Alexandria, Egypt.

Funding Open access funding provided by The Science, Technology & Innovation Funding Authority (STDF) in cooperation with The Egyptian Knowledge Bank (EKB).

Data availability The data that support the findings of this study are available from the corresponding author upon reasonable request.

Open Access This article is licensed under a Creative Commons Attribution 4.0 International License, which permits use, sharing, adaptation, distribution and reproduction in any medium or format, as long as you give appropriate credit to the original author(s) and the source, provide a link to the Creative Commons licence, and indicate if changes were made. The images or other third party material in this article are included in the article's Creative Commons licence, unless indicated otherwise in a credit line to the material. If material is not included in the article's Creative Commons licence and your intended use is not permitted by statutory regulation or exceeds the permitted use, you will need to obtain permission directly from the copyright holder. To view a copy of this licence, visit <http://creativecommons.org/licenses/by/4.0/>.

References

1. I. Qasim, M. Waqee-ur-Rehman, M. Mumtaz, G. Hussain, K. Nadeem, N.A. Khan, Role of anti-ferromagnetic Cr nanoparticles in $CuTi-1223$ superconducting matrix. *J. Alloys Compd.* **649**, 320 (2015)
2. M.M. Elokr, R. Awad, A. Abd El-Ghany, A. Abou Shama, A. Abd El-wanis, Effect of nano-sized ZnO on the physical properties of $(Cu_{0.5}Ti_{0.25}Pb_{0.25})Ba_2Ca_2Cu_3O_{10-\delta}$. *J. Supercond. Novel Magn.* **24**, 1345 (2011)
3. A. Jabbar, I. Qasim, M. Waqee-ur-Rehman, M. Zaman, K. Nadeem, M. Mumtaz, Structural and superconducting properties of $(Al_2O_3)_y/CuTi-1223$ composites. *J. Electron. Mater.* **44**, 110 (2015)
4. M. Imran, M. Zareef Khan, M. Waqee-Ur-Rehman, A. Ullah, S. Ahmed, K. Nadeem, M. Mumtaz, Role of Co_3O_4 nanoparticles addition in in-field superconducting properties of $CuTi-1223$ phase. *J. Low Temp. Phys.* **200**, 152 (2020)
5. N.H. Mohammed, A.I. Abou-Aly, I.H. Ibrahim, R. Awad, M. Rekaby, Effect of nano-oxides addition on the mechanical properties of $(Cu_{0.5}Ti_{0.5})-1223$ phase. *J. Supercond. Novel Magn.* **24**, 1463 (2011)
6. A. Srour, W. Malaeb, M. Rekaby, R. Awad, Mechanical properties of the $(BaSnO_3)_x/Cu_{0.5}Ti_{0.5}Ba_2Ca_2Cu_3O_{10-\delta}$ superconductor phase. *Phys. Scr.* **92**, 104000 (2017)
7. R. Awad, Study of the influence of MgO nano-oxide addition on the electrical and mechanical properties of $(Cu_{0.25}Ti_{0.75})-1234$ superconducting phase. *J. Supercond. Novel Magn.* **21**, 461 (2008)
8. O. Uzun, U. Kölemen, S. Çelebi, N. Güçlü, Modulus and hardness evaluation of polycrystalline superconductors by dynamic microindentation technique. *J. Eur. Ceram. Soc.* **25**, 969 (2005)
9. M. Muralidhar, K. Narasimha Reddy, V. Hari Babu, Microhardness studies on $BiCaSrCuO$ (2122) superconducting single crystals and pellets. *Phys. Status Solidi (a)* **126**, 115 (1991)
10. C. Veender, V.R. Dumke, M. Nagabhooshanam, Hardness and elastic moduli of $Bi_2-xPbxCa_2Sr_2Cu_3O_y$ superconductors. *Phys. Status Solidi (a)* **144**, 299 (1994)
11. A. Tampieri, G. Celotti, S. Guicciardi, C. Melandri, Microstructural and mechanical characterization of bulk BSCCO (2223) superconductor. *Mater. Chem. Phys.* **42**, 188 (1995)
12. M. Ionescu, B. Zeimet, S.X. Dou, Microhardness anisotropy of $Bi-2212$ crystals. *Phys. C Supercond.* **306**, 213 (1998)
13. S.M. Khalil, Effects of optimum annealing time on superconducting properties of $Bi_2-xPbSr_2Ca_2Cu_3O_y$ system. *Phys. Status Solidi (a)* **178**, 731 (2000)
14. E. Asikuzun, O. Ozturk, H.A. Cetinkara, G. Yildirim, A. Varilci, M. Yılmazlar, C. Terzioğlu, Vickers hardness measurements and some physical properties of Pr_2O_3 doped $Bi-2212$ superconductors. *J. Mater. Sci. Mater. Electron.* **23**, 1001 (2012)
15. A. Ghattas, M. Annabi, M. Zouaoui, F. Ben Azzouz, M. Ben Salem, Flux pinning by Al-based nano particles embedded in polycrystalline $(Bi, Pb)-2223$ superconductors. *Phys. C Supercond. Appl.* **468**, 31 (2008)
16. B. Zhao, X. Wan, W. Song, Y. Sun, J. Du, Nano-MgO particle addition in silver-sheathed $(Bi, Pb) 2Sr_2Ca_2Cu_3O_x$ tapes. *Phys. C Supercond.* **337**, 138 (2000)
17. W. Kong, R. Abd-Shukur, Enhanced electrical transport properties of nano $NiFe_2O_4$ -added $(Bi_{1.6}Pb_{0.4})Sr_2Ca_2Cu_3O_{10}$ superconductor. *J. Supercond. Novel Magn.* **23**, 257 (2010)
18. B.A. Albiss, I.M. Obaidat, M. Gharaibeh, H. Ghamlouche, S.M. Obeidat, Impact of addition of magnetic nanoparticles on vortex pinning and microstructure properties of $Bi-Sr-Ca-Cu-O$ superconductor. *Solid State Commun.* **150**, 1542 (2010)
19. S.M. Khalil, Enhancement of superconducting and mechanical properties in BSCCO with Pb additions. *J. Phys. Chem. Solids* **62**, 457 (2001)
20. X. Liu, H. Li, M. Zhan, A review on the modeling and simulations of solid-state diffusional phase transformations in metals and alloys. *Manuf. Rev.* **5**, 10 (2018)
21. A.M. Roy, Formation and stability of nanosized, undercooled propagating intermediate melt during $\beta \rightarrow \delta$ phase transformation in HMX nanocrystal. *Europhys. Lett.* **133**, 56001 (2021)
22. A.M. Roy, Energetics and kinematics of undercooled nonequilibrium interfacial molten layer in cyclotetramethylene-tetranitramine crystal. *Phys. B Condens. Matter* **615**, 412986 (2021)
23. E. Guilmeau, B. Andrzejewski, J.G. Noudem, The effect of MgO addition on the formation and the superconducting properties of the $Bi2223$ phase. *Phys. C Supercond.* **387**, 382 (2003)
24. A. Leenders, M. Mich, H.C. Freyhard, Influence of thermal cycling on the mechanical properties of VGF melt-textured YBCO. *Phys. C Supercond.* **279**, 173 (1997)
25. S. Farhat, M. Rekaby, R. Awad, Synthesis and characterization of Er-doped nano ZnO samples. *J. Supercond. Novel Magn.* **31**, 3051 (2018)
26. K. Sangwal, B. Surowska, Study of indentation size effect and microhardness of $SrLaAlO_4$ and $SrLaGaO_4$ single crystals. *Mater. Res. Innov.* **7**, 91 (2003)
27. R. Tickoo, R.P. Tandon, K.K. Bamzai, P.N. Kotru, Microindentation studies on samarium-modified lead titanate ceramics. *Mater. Chem. Phys.* **80**, 446 (2003)
28. C. Hays, E.G. Kendall, An analysis of Knoop microhardness. *Metallography* **6**, 275 (1973)
29. N. Gane, F.P. Bowden, Microdeformation of solids. *J. Appl. Phys.* **39**, 1432 (1968)
30. H.T. Rahal, R. Awad, A.M. Abdel Gaber, M. Roumie, Superconducting and mechanical properties of the bulk $(SnO_2)_x(Bi_{1.6}Pb_{0.4})Sr_2Ca_2Cu_3O_{10-\delta}$ prepared at different sintering times. *J. Supercond. Novel Magn.* **30**, 1971 (2017)
31. V.R. Howes, Mechanical indentation testers designed to allow the continuous microscopic observation of the controlled indentation of transparent materials. *J. Phys. E Sci. Instrum.* **15**, 1308 (1982)

32. M.M. Barakat, A.I. Abou-Aly, R. Awad, N.S. Aly, S. Ibrahim, Mechanical properties of $Y_{3-x}Nd_xBa_{5-x}Ca_xCu_8O_{18-\delta}$ samples. *J. Alloys Compd.* **652**, 158 (2015)
33. L. Hong, R.C. Bradt, The microhardness indentation load/size effect in rutile and cassiterite single crystals. *J. Mater. Sci.* **28**, 917 (1993)
34. J. Gong, H. Miao, Z. Zhao, Z. Guan, Load-dependence of the measured hardness of Ti (C, N)-based cermets. *Mater. Sci. Eng. A* **303**, 179 (2001)
35. J.B. Quinn, G.D. Quinn, Indentation brittleness of ceramics: a fresh approach. *J. Mater. Sci.* **32**, 4331 (1997)
36. O. Ozturk, E. Asikuzun, A.T. Tasci, T. Gokcen, H. Ada, H. Koray, S. Cavdar, Comparison of Vickers microhardness of undoped and Ru doped BSCCO glass ceramic materials. *J. Mater. Sci. Mater. Electron.* **29**, 3957 (2018)
37. N.H. Mohammed, A.I. Abou-Aly, R. Awad, I.H. Ibrahim, M. Roumie, M. Rekaby, Mechanical and electrical properties of $(Cu_{0.5}Ti_{0.5})-1223$ phase added with nano- Fe_2O_3 . *J. Low Temp. Phys.* **172**, 234 (2013)
38. W. Zewen, J. Wanqi, Microhardness of $Hg_{1-x}Mn_xTe$. *Mater. Sci. Eng. A* **452**, 508 (2007)

Publisher's Note Springer Nature remains neutral with regard to jurisdictional claims in published maps and institutional affiliations.