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Influence of cobalt addition and calcination temperature on the physical properties of BaFe₁₂O₁₉ hexaferrites nanoparticles

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Abstract

Nano-scale particles of pure Barium hexaferrite 'BaFe₁₂O₁₉' and Cobalt added Barium hexaferrite 'Co_xBaFe₁₂O₁₉', with $x = 0.04, 0.06$ and 0.1 wt%, were successfully synthesized by the chemical co-precipitation method. The synthesized powder was subjected to different calcination temperatures ($T = 850\text{ }^{\circ}\text{C}, 900\text{ }^{\circ}\text{C}, 950\text{ }^{\circ}\text{C}$ and $1050\text{ }^{\circ}\text{C}$). X-ray powder diffraction (XRD) clarified that nearly single phase of BaFe₁₂O₁₉ with tiny traces of Fe₂O₃ phase were obtained when the precursor was calcined at $1050\text{ }^{\circ}\text{C}$ for 2 h. The lattice parameters and unit cell volume were almost unchanged with either Cobalt addition or calcination temperatures. From Debye–Scherrer equation, the crystallite size (D) was found to gradually increase with increasing calcination temperature to reach its maximum values for samples calcined at $1050\text{ }^{\circ}\text{C}$. The formation of Barium hexaferrite phase was also confirmed from Fourier transform infrared (FTIR) spectra through the existence of strong absorption peaks that appeared between 581 cm^{-1} and 435 cm^{-1} . The morphology and grain size of the samples were examined using transmission electron microscopy (TEM) technique. Optical properties of the samples were studied through ultraviolet 'UV' visible spectroscopy. The optical band gap (E_g) of the samples was obtained from Tauc relation as function of Cobalt addition (x) and calcination temperature (T). Finally, the mechanical properties were examined using Vickers microhardness. The microhardness data revealed that the samples exhibited reverse indentation size effect (RISE). The Elastic modulus (E) and yield strength (Y) for the prepared samples were calculated, in accordance with Vickers microhardness, as function of Cobalt addition. Furthermore, the indentation size effect ISE was analyzed using indentation induced cracked model (IIC). The IIC model was found to be a suitable model for describing the microhardness results of the prepared samples. Time dependent Vickers microhardness was done through indentation creep test at different dwell time ($t = 10, 20, 30, 40$ and 50 s) and constant applied loads ($F = 0.98, 4.90$ and 9.80 N). Results clarified that the specimens revealed grain boundary sliding together with dislocation climbs at small loads and a dislocation creep in the operating creep process for greater loads.

1. Introduction

Hexaferrites are wide category of ferromagnetic oxide materials that crystallize in complex hexagonal structure with space group $P6_3/mmc$ [1]. Six types of hexaferrites are classified and symbolized as M, Y, W, Z, X and U denoting BaFe₁₂O₁₉, Ba₂Me₂Fe₁₂O₂₂, BaMe₂Fe₁₆O₂₇, Ba₃Me₂Fe₂₄O₄₁, Ba₂Me₂Fe₂₈O₄₆ and Ba₄Me₂Fe₃₆O₆₀, respectively. In the chemical formulae of hexaferrites Me is a divalent metal ion (such as: Co, Ni, Zn... etc) and Ba can be replaced by Pb or Sr [2]. The unit cell of Barium-based hexaferrites consists of specific stacking sequence of close-packed oxygen layers with Barium ions partially replacing Oxygen ions at particular positions, and the small metallic ions occupying interstitial positions [3]. Among all Barium hexaferrites, the M-type Barium hexaferrite 'BaFe₁₂O₁₉' is found to be scientifically and technologically promising due to its amazing

properties and its numerous applications. $\text{BaFe}_{12}\text{O}_{19}$ characterizes by its high magnetic anisotropy field, high coercive force and high Curie temperature. Also, it has an excellent chemical stability and corrosion resistivity [4, 5]. On the technological side, it has been used to manufacture permanent magnets, magnetic recording media, microwave devices and circuits, magneto-optic media, signal processing devices, telecommunications devices, supercapacitors and microwave filters [6–13]. Synthesis of nano-scale particles of $\text{BaFe}_{12}\text{O}_{19}$ can be achieved through different methods including: co-precipitation [14, 15], sol–gel [16, 17], carbon combustion [18], citrate auto-combustion [19] and hydrothermal [20].

The effect of substitution/addition on the physical properties of $\text{BaFe}_{12}\text{O}_{19}$ has been recorded in many literatures. Substitutions can be done by the partial replacement of Fe^{3+} ions with other cations such as Mn–Sn, Co–Sn and Mn–Co–Sn [21], Co [22, 23] and Ni [24]. Moreover, substitutions can be done by the partial replacement of Ba^{2+} ions with other cations as reported by Taufeeq *et al* [25] for studying the structural and thermal properties of pure $\text{BaFe}_{12}\text{O}_{19}$ and Sr doped $\text{Ba}_{0.9}\text{Sr}_{0.1}\text{Fe}_{12}\text{O}_{19}$ nanostructure. They concluded that Sr-doping sustains the hexagonal crystal structure of Ba hexaferrites. Also, they observed a decrement in the crystallite size and an increment in the lattice parameters. The thermal property studied by TGA showed weight loss as a function of both temperature and Sr doping. Rusianto *et al* [26] studied the magnetic and mechanical properties of $\text{Sr}_x\text{Ba}_{1-x}\text{Fe}_{12}\text{O}_{19}$ (with $x = 0.00, 0.25, 0.50, 0.75$ and 1.00) synthesized with conventional method. They found that at sintering temperature 1100°C the saturation magnetization M_s and the retentivity M_r decreased with x , while the coercivity H_c increased. The flexural strength and the Vickers microhardness are found to increase with increasing sintering temperature. The addition of Bismuth [27] increased the density and improved the synthesis rate of $\text{BaFe}_{12}\text{O}_{19}$. Also, it is found that the addition of Si reduces the grain size and increases the coercivity [28] of $\text{BaFe}_{12}\text{O}_{19}$. Setiadi *et al* [29] investigated the microstructures, physical and magnetic properties of $\text{BaFe}_{12}\text{O}_{19}$ added with Al_2O_3 -MnO (0.12, 0.24 and 0.42 wt%). Particle size analyzer measurements showed that the particle sizes of $\text{BaFe}_{12}\text{O}_{19}$ decrease as the additive concentrations of Al_2O_3 -MnO increase. Moreover, the hardness of the $\text{BaFe}_{12}\text{O}_{19}$ is found to increase with Al_2O_3 -MnO addition and this was related to the increase of bulk density values. There was a decrease in the magnetic flux, saturation and remanence values of $\text{BaFe}_{12}\text{O}_{19}$ with the addition of Al_2O_3 -MnO concentrations.

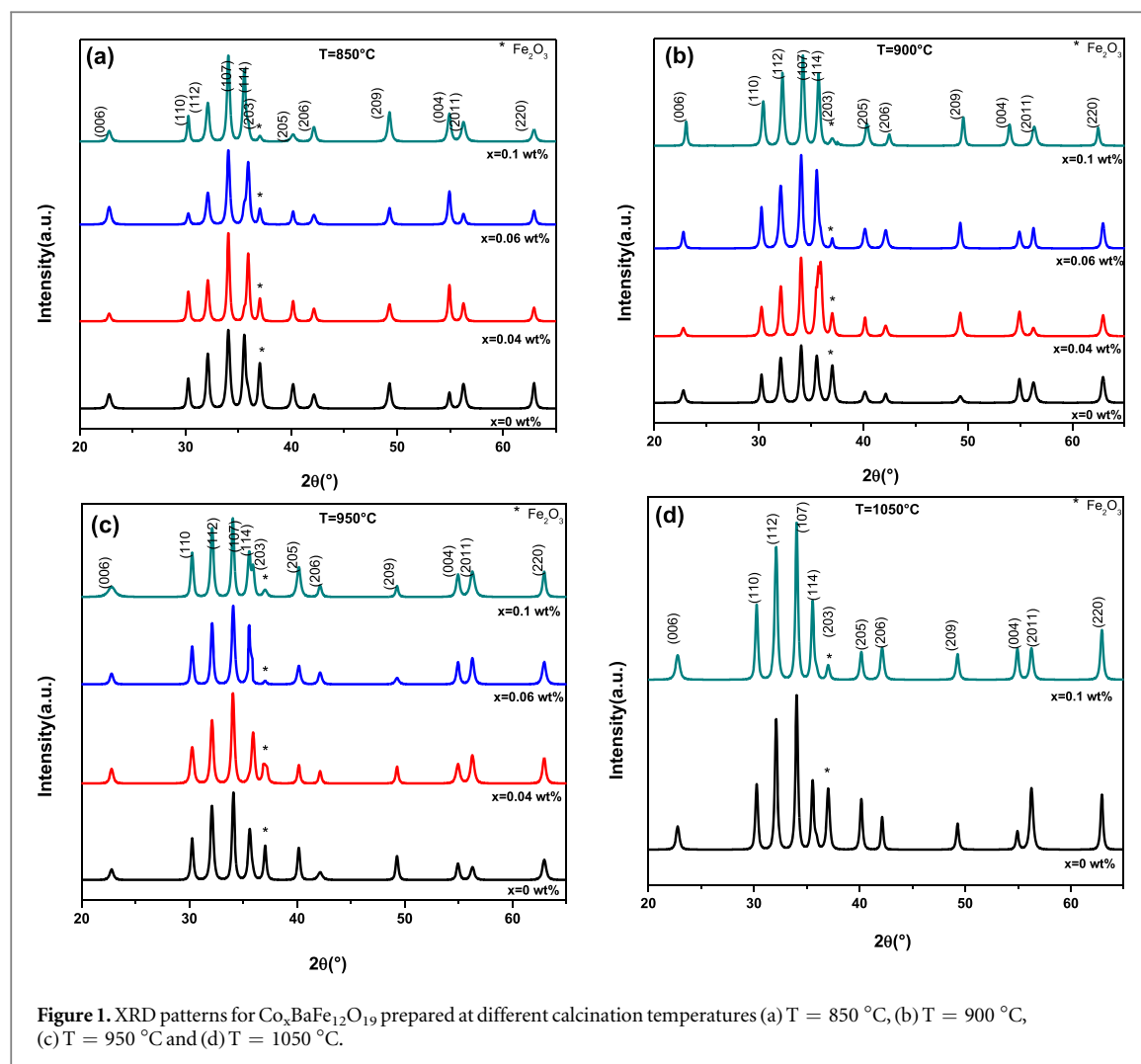
The magnetic properties of $\text{BaFe}_{12}\text{O}_{19}$ were extensively studied, since their discovery, in many literatures [26–29]. On the contrary few data are available on their mechanical properties. One of the drawbacks of hexaferrites is their ceramic nature that makes them not flexible and moldable. However, for some applications for instance; in magnetomechanical transducers and in surface-mounted devices (SMD) such as inductors and transformer cores [30], materials must possess appropriate mechanical strength requiring precise control of the microstructure. Therefore, it is of major interest to study the mechanical properties of hexaferrites, which can give a broad insight into their microstructure. Vickers microhardness regarded as a quick and non-destructive test to extract information on the mechanical behavior of materials such as; resistance to plastic deformation, modulus of elasticity, brittleness index, yields strength, ductility, fractures toughness and cracking temperature.

In this study, the influence of Cobalt addition and the calcination temperatures on the mechanical and optical properties of Barium hexaferrites was investigated. The incorporation of Cobalt in $\text{BaFe}_{12}\text{O}_{19}$ matrix and elevated calcination temperature may enhance the microstructure and in turn the mechanical strength of $\text{BaFe}_{12}\text{O}_{19}$ samples.

2. Materials and methods

2.1. Synthesis procedure

Nano-scale particles of pure Barium hexaferrite ' $\text{BaFe}_{12}\text{O}_{19}$ ' and Cobalt added Barium hexaferrite ' $\text{Co}_x\text{BaFe}_{12}\text{O}_{19}$ ', with $x = 0.04, 0.06$ and 0.10 wt%, were synthesized by the chemical co-precipitation method. The synthesized powder was subjected to different calcination temperatures ($T = 850^\circ\text{C}, 900^\circ\text{C}, 950^\circ\text{C}$ and 1050°C). Stoichiometric amounts of Iron (III) chloride hexahydrate ($\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$), Cobalt Chloride (CoCl_2) and Barium Chloride Dihydrate ($\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$) were used to prepare aqueous solutions in appropriate amounts of distilled water. A solution of Poly vinyl pyrrolidone (PVP) was prepared by dissolving 4 g of this polymer in 100 ml distilled water and was heated at 80°C for 15 min. Both solutions were mixed and kept under constant stirring by using magnetic stirrer for few minutes before the addition of NaOH. 4 M of NaOH was added with continuous stirring till the pH of the solution becomes 13. After that the solution was heated for 2 h at 60°C with continuous stirring to transform the hydroxide into ferrites. The precipitate was washed several times till the pH become 7 and then was dried at 100°C for 18 h. After that, calcination at different temperatures ($T = 850^\circ\text{C}, 900^\circ\text{C}, 950^\circ\text{C}$ and 1050°C) was performed for 2 h using tube furnace.



2.2. Characterization and measurements

Room temperature x-ray powder diffraction was performed using advance powder diffractometer with Cu target ($\lambda = 1.54056 \text{ \AA}$) in 2θ range of 10° to 80° . FTIR analysis was carried out using FTIR Nicolet iS5- Thermoscientific. JEOL transmission microscope JEM-100CX is used to provide morphology information for the synthesized samples. At room temperature, UV-visible measurements have been taken using spectrophotometer V-670 ultraviolet-near infrared (NIR). Vickers microhardness was measured with a digital microhardness tester model MHVD-1000IS. Vickers indenter is in the form of square-pyramid diamond. On a clean sample surface, the indenter was applied under test loads varying from 0.49 N to 9.80 N for dwell time 60 s. To assure accuracy of microhardness data, the average value of 3 indentations at different locations on the sample surface was taken for each specific load. Vickers indentation marks left on the sample surface after offloading were nearly square in shape with two diagonals. A calibrated micrometer eye-piece mounted to the microscope was employed to estimate the length of the two diagonals after offloading. The diagonal lengths of indentation were estimated with precision of $\pm 0.1 \mu\text{m}$. At different dwell times ($t = 10, 20, 30, 40$ and 50 s) and for constant loads of 0.98, 4.90 and 9.80 N, indentation creep test was carried out for the prepared samples.

3. Results and discussion

3.1. XRD

Figures 1(a)–(d) shows XRD patterns for $\text{Co}_x\text{BaFe}_{12}\text{O}_{19}$ nanoparticles prepared at different calcination temperatures $T = 850^\circ\text{C}$, 900°C , 950°C and 1050°C , respectively. To be noted that figure 1(d) shows XRD patterns only for two samples of $\text{Co}_x\text{BaFe}_{12}\text{O}_{19}$ with $x = 0$ and 0.1 wt% prepared at calcination temperature $T = 1050^\circ\text{C}$. The observed peaks in figures 1(a)–(d) are well indexed by the hexagonal indices of $\text{BaFe}_{12}\text{O}_{19}$ (ICDD No-840757) with space group $P6_3/\text{mmc}$. Minor peak of Fe_2O_3 phase is also presented in the patterns at $2\theta \approx 37^\circ$. Ding *et al* [31] reported from Mössbauer spectra that Fe_2O_3 is usually associating the formation of

Table 1. Phase percentage (BaFe₁₂O₁₉), lattice parameters (a and c), unit cell volume (V), crystalline size (D) and grain size (D_{TEM}) of Co_xBaFe₁₂O₁₉ nanoparticles with (0 ≤ x ≤ 0.1) at different calcination temperatures.

T(°C)	x (wt%)	BaFe ₁₂ O ₁₉ %	a(Å)	c(Å)	V (Å ³)	D (nm)	D _{TEM} (nm)
850	0	89.4	5.87	23.11	690.3	45.42	65.04
	0.04	90.26	5.89	23.10	692.7	46.38	95.02
	0.06	90.43	5.87	23.13	690.9	51.5	94.04
	0.1	90.45	5.87	23.11	690.3	53.494	87.81
900	0	90.36	5.87	23.14	692.6	51.5	90.20
	0.04	90.43	5.88	23.21	693.6	55.57	98.75
	0.06	90.83	5.87	23.16	696.9	51.42	101.19
	0.1	90.84	5.87	23.12	693.0	54.98	80.05
950	0	91.00	5.87	23.13	692.7	58.76	121.57
	0.04	91.01	5.87	23.13	693.3	52.02	167.80
	0.06	91.65	5.878	23.16	693.9	54.60	78.59
	0.1	91.62	5.87	23.12	692.0	57.54	71.81
1050	0	91.69	5.87	23.12	691.7	58.86	121.25
	0.1	91.83	5.87	23.13	692.3	60.57	110.54

Barium hexaferrite. The phase percentage of BaFe₁₂O₁₉ is calculated and is listed in table 1. It is clear for all the samples that the phase percentage of BaFe₁₂O₁₉ is enhanced with increasing both Co-addition and calcination temperature. For instance, samples prepared at T = 850 °C their BaFe₁₂O₁₉ phase percentage increases from 89.4% to 90.45% as Co- addition (x) increases from 0 to 0.1 wt%. Moreover, the percentage of pure BaFe₁₂O₁₉ phase increases from 89.4% to 91.69% as T increases from 850 °C to 1050 °C. This means that, increasing Cobalt content and calcination temperature decreases the percentage of the intermediate phases and increases the percentage of barium hexaferrite phase. Another observation is that the calcination temperature is found to be slightly more effective in improving the phase percentage of BaFe₁₂O₁₉ than Co-addition. Furthermore, the optimum calcination temperature for nearly single phase of BaFe₁₂O₁₉ with minor traces of Fe₂O₃ is observed for samples calcined at T = 1050 °C. Similar results were reported by Packiaraj *et al* [32]. They concluded that by increasing the annealing temperature the intermediate phases such as γ-Fe₂O₃, α-Fe₂O₃ and BaFe₂O₄ decreased whereas BaFe₁₂O₁₉ phase increased and appeared as a major phase at 950 °C. Zhong *et al* [33] reported about the fact that the appearance of Fe₂O₃ with orthorhombic hexahedron structure prevents the formation of BaFe₁₂O₁₉ hexagonal structure at low temperature below 750 °C.

Lattice parameters (a and c) and unit cell volume (V) are calculated according to the following equations [34, 35]

$$\frac{1}{d^2} = \frac{4}{3} \times \frac{h^2 + hk + k^2}{a^2} + \frac{l^2}{c^2}, \quad (1)$$

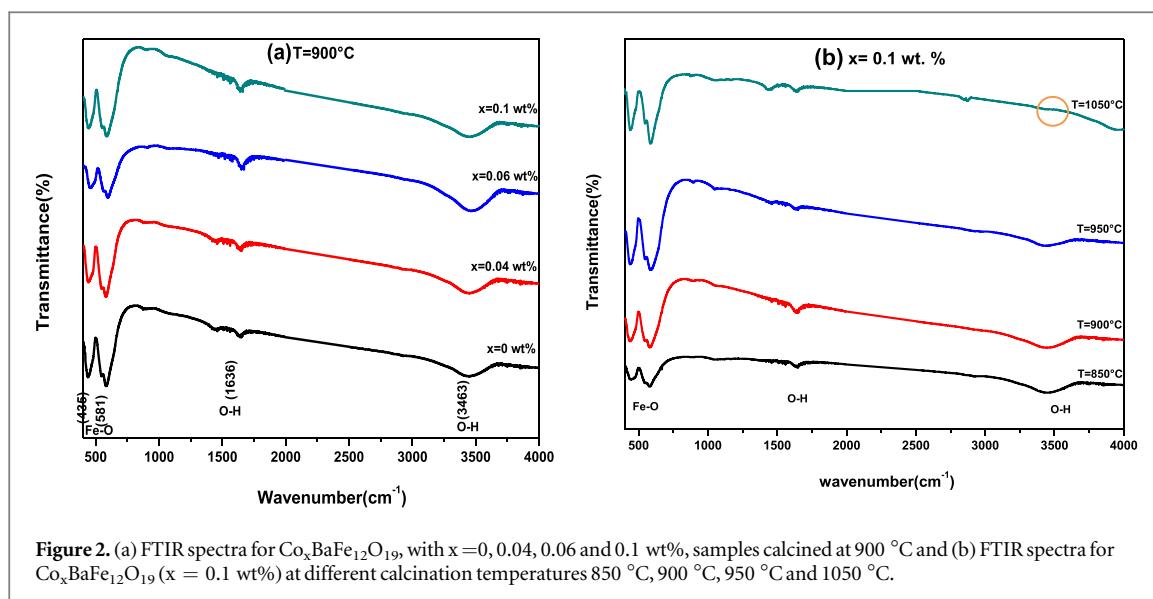
$$V = \frac{\sqrt{3}}{2} a^2 c, \quad (2)$$

where *d* is the interplanar distance and (*h k l*) are the Miller indices. The average crystallite size of the samples was estimated using Debye–Scherrer equation;

$$D = \frac{S\lambda}{\beta_{hkl} \cos \theta} \quad (3)$$

where *D* is the average crystallite size in nanometers, *S* is the shape factor and it is equal to 0.9, *λ* is the wavelength of the x-ray radiation, *β_{hkl}* is the peak width at half maximum intensity and *θ* is the peak position.

The values of a, c, V and D for Co_xBaFe₁₂O₁₉ samples prepared at different calcination temperatures (T = 850 °C, 900 °C, 950 °C and 1050 °C) are listed in table 1. It is clear, from table 1, that the lattice parameters and unit cell volume are almost unchanged with either Cobalt addition or calcination temperature. This indicates that Cobalt occupies only interstitial places in Barium hexaferrite matrix without entering the structure and that the calcination temperature has no effect in changing the crystal structure of the phase. The crystallite size *D* varies directly with increasing the Co-content (*x*) for samples prepared at T = 850 °C and 1050 °C while it has unsystematic variation with *x* for samples prepared at T = 900 °C and 950 °C. Also, it can be seen from table 1 that the crystallite size (*D*) is gradually increased with increasing calcination temperature to reach its maximum values for samples calcined at 1050 °C. This result is consistent with the XRD peaks width which becomes narrower with increasing calcination temperature, indicating that the average crystalline size of synthesized ferrites is gradually increased.



3.2. FTIR spectroscopy

To confirm hexaferrite phase formation, the prepared samples were analyzed via Fourier transformed Infrared (FTIR) analysis in wavenumber range of 400 cm^{-1} to 4000 cm^{-1} . Figure 2(a) shows room temperature FTIR spectra for $\text{Co}_x\text{BaFe}_{12}\text{O}_{19}$, with $x = 0, 0.04, 0.06$ and 0.1 wt%, samples calcined at 900°C . The strong absorption peaks between 581 cm^{-1} and 435 cm^{-1} confirm the formation of hexaferrite phase [36, 37]. Peaks at 435 cm^{-1} and 581 cm^{-1} are assigned to magnetite stretching vibration bonds of Fe–O at tetrahedral and octahedral sites [38]. Absorption peak centered at 3463 and 1636 cm^{-1} are attributed to stretching and bending vibration of H_2O caused by adsorbed water or humidity which shows that the surface of the samples contain active O–H groups [39]. It is obvious, from figure 2(a), that the peaks position of the pure sample ($x = 0$ wt%) is not altered or shifted after Cobalt additions. This reflects that $\text{BaFe}_{12}\text{O}_{19}$ structure was not perturbed by the presence of Cobalt in its matrix, which is consistent with XRD results. Moreover, the influence of calcination temperature on $\text{BaFe}_{12}\text{O}_{19}$ structure is displayed in figure 2(b) for $\text{Co}_x\text{BaFe}_{12}\text{O}_{19}$ with $x = 0.1$ wt%. It is obvious, from figure 2(b), that the peaks position of the sample is slightly affected by increasing the calcination temperature from 850°C to 1050°C . For instance, the peak that defines the stretching vibration bonds of Fe–O at the tetrahedral site changed from 435.66 cm^{-1} to 435.35 cm^{-1} as the calcination temperature increased from 850°C to 1050°C . Furthermore, peak attributed to the bending of H_2O varied from 1636.04 cm^{-1} to 1636.32 as T increased from 850°C to 1050°C . This behavior indicates that there is no change occurs in the bond strength of magnetite and H_2O at these calcination temperatures [40]. However, at 1050°C there is a disappearance of the stretching vibration peak of H_2O at 3463 cm^{-1} . This disappearance at high temperature could be attributed to vaporization process that could eliminate the active O–H groups trapped at the sample surface [40].

3.3. TEM

The morphology and average grain size of the prepared samples were examined using TEM technique. Figures 3(a)–(c) shows TEM images coupled with their grain size histograms for $\text{Co}_x\text{BaFe}_{12}\text{O}_{19}$ samples with ($x = 0$ wt%, $T = 850^\circ\text{C}$), ($x = 0.1$ wt%, $T = 900^\circ\text{C}$) and ($x = 0.1$ wt%, $T = 1050^\circ\text{C}$), respectively. The data are fitted to the Gaussian distribution and the peak values of the fit are taken as the average grain size of the selected samples. The average grain sizes D_{TEM} for all the synthesized samples are listed in table 1. It is clear, from table 1, that the average grain sizes increase with increasing the calcination temperature and that they range from 70 to 121 nm . TEM micrographs showed that the samples composed of agglomerated grains that form clusters of different sizes and shapes. Samples possess spherical together with hexagonal shaped grains. Similar morphology was observed for Barium hexaferrite with diameter 50 – 70 nm by Jurek *et al* [41]. Also, it is noticed that the hexagonal shaped grains becomes clearly defined as the Cobalt content and calcination temperature are increased to 0.1 wt% and 1050°C , respectively. This result is mainly due to the increase in the phase formation of $\text{BaFe}_{12}\text{O}_{19}$ which is enhanced at high Cobalt content (x) and elevated calcination temperature. Therefore, TEM result is in agreement with XRD result and phase percentage calculation. Similar result was found by Mehmedi *et al* [42] for Ba and Sr hexaferrites added by B_2O_3 . They concluded that at 1000°C and with B_2O_3 addition the hexagonal grain geometry of the samples starts to be well shaped compared to other samples calcined at lower temperatures and without B_2O_3 addition.

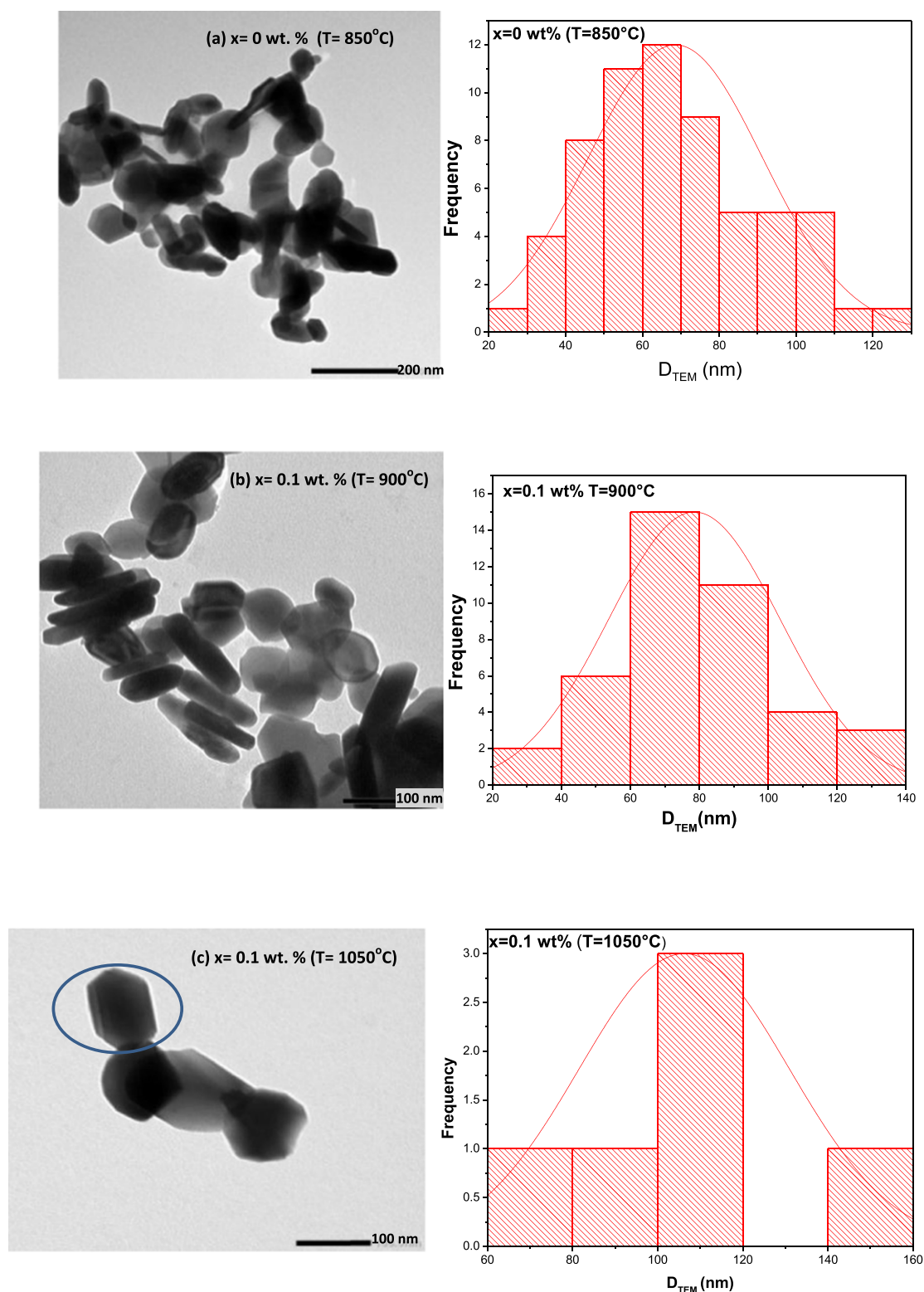


Figure 3. TEM images coupled with their grain size histograms fitted to Gaussian distribution for $\text{Co}_x\text{BaFe}_{12}\text{O}_{19}$ with (a) $x = 0$ wt% ($T = 850^\circ\text{C}$), (b) $x = 0.1$ wt% ($T = 900^\circ\text{C}$) and (c) $x = 0.1$ wt% ($T = 1050^\circ\text{C}$).

3.4. Optical measurements (UV-visible spectroscopy)

Figure 4(a) displays the UV-visible absorbance spectra of $\text{Co}_x\text{BaFe}_{12}\text{O}_{19}$ as a function of Co addition (x) calcined at $T = 850^\circ\text{C}$. It is clear that, the absorbance peak is slightly shifted to higher wavelengths with increasing Co addition. Agrawal *et al* [43] reported similar results for Ca and Ni doped Barium hexaferrite. This slight shift could be explained in terms of the s, p-d spin-exchange interactions between delocalized s- or p-type band electrons of Fe and O atoms, respectively and localized d-electrons of transition metal Co [44]. Figure 4(b) shows the UV-visible absorbance spectra of $\text{Co}_x\text{BaFe}_{12}\text{O}_{19}$ with $x = 0.1$ wt% as function of calcination temperatures

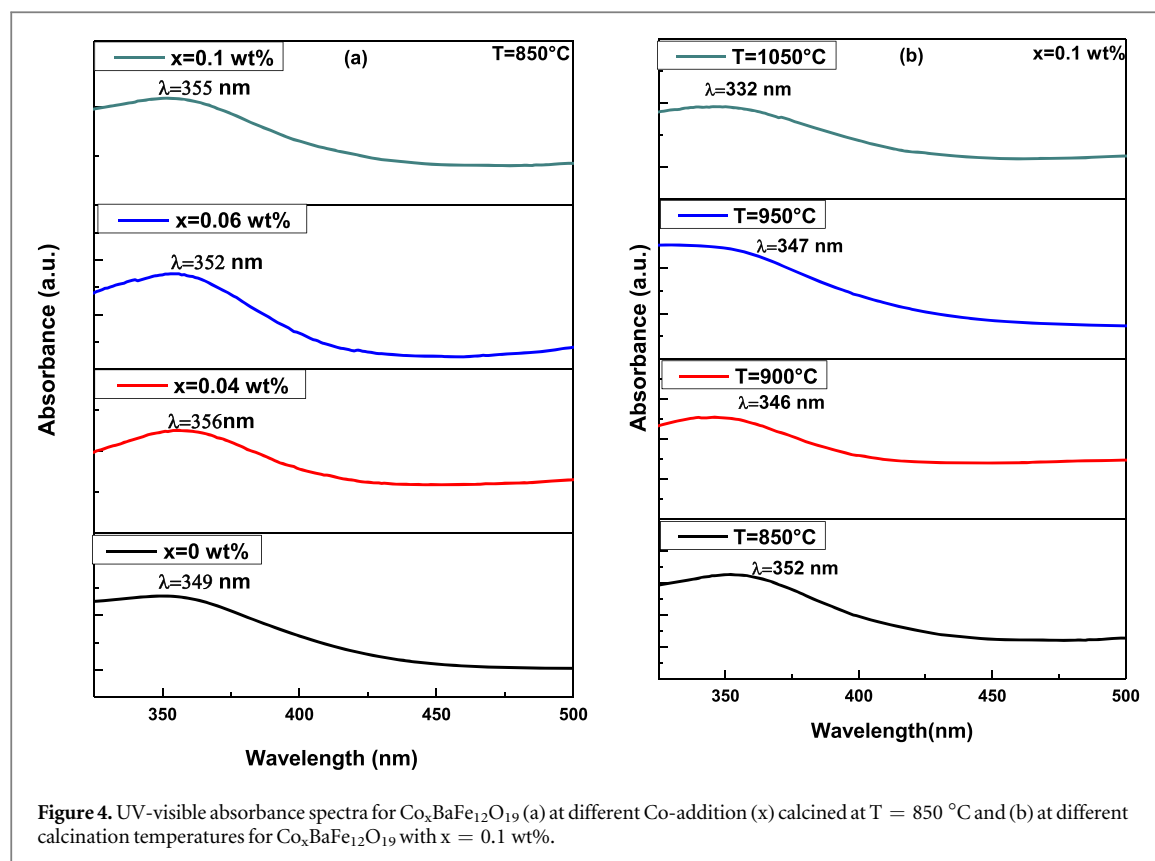


Figure 4. UV-visible absorbance spectra for $\text{Co}_x\text{BaFe}_{12}\text{O}_{19}$ (a) at different Co-addition (x) calcined at $T = 850^\circ\text{C}$ and (b) at different calcination temperatures for $\text{Co}_x\text{BaFe}_{12}\text{O}_{19}$ with $x = 0.1$ wt%.

$T = 850^\circ\text{C}$, 900°C , 950°C and 1050°C . The absorbance peak around wavelength of 352 nm for $\text{Co}_x\text{BaFe}_{12}\text{O}_{19}$ with $x = 0.1$ wt% is slightly shifted to lower wavelengths with increasing the calcination temperature from 850 to 1050°C . In general, the absorbance shift depends mainly on various factors such as band gap, grain size, impurities, oxygen deficiency and surface effects.

In the optical region, when electromagnetic waves fall on the samples it causes the valance band electrons to absorb energy and to rise to higher energy level. This absorbed band gap energy is calculated from Tauc relation [45]:

$$\alpha h\nu = B(h\nu - E_g)^n, \quad (4)$$

where α is the absorption coefficient, B is a constant, $h\nu$ is the energy of incident photons, and $n = 1/2$ for allowed direct transitions. E_g is the optical band gap.

Figure 5 shows the plot of $(\alpha h\nu)^2$ versus photon energy $h\nu$, according to Tauc relation, for pure $\text{BaFe}_{12}\text{O}_{19}$ calcined at $T = 850^\circ\text{C}$. The optical band gap E_g for pure and Co added $\text{BaFe}_{12}\text{O}_{19}$ samples are obtained from the extrapolation of $(\alpha h\nu)^2$ to zero and tabulated in table 2. It is found that the band gap of pure $\text{BaFe}_{12}\text{O}_{19}$ is 4.25 eV, 4.05 eV, 3.83 eV and 3.71 eV calcined at 850°C , 900°C , 950°C and 1050°C , respectively. Karmakar et al [45] estimated that the band gap for Barium hexaferrite is around 3.18 eV which is lower than those recorded for our pure $\text{BaFe}_{12}\text{O}_{19}$ calcined at different temperatures. It is clear that, the values of the energy bandgap decreases with Co addition from 4.25 eV to 3.95 eV as x increases from 0 wt% to 0.1 wt% at $T = 850^\circ\text{C}$. Same results were obtained for the other temperatures ($T = 900^\circ\text{C}$, $T = 950^\circ\text{C}$ and $T = 1050^\circ\text{C}$) where the E_g decreases with Co addition. As a result, both Co addition and calcination temperatures decrease the band gap of barium hexaferrite nanoparticles. The reduction in optical band gap E_g values could be attributed to the enhancement in the samples grain size [45].

3.5. Vickers microhardness, Elastic modulus and yield strength

Figures 6(a)–(c) displays the variation of Vickers microhardness (H_v) with the applied test load (F), at dwell time $t = 60$ s, for $\text{Co}_x\text{BaFe}_{12}\text{O}_{19}$ samples calcined at 900°C , 950°C and 1050°C , respectively. The inset of figure 6(a) depicts Vickers indentation marks taken on the pure $\text{Co}_x\text{BaFe}_{12}\text{O}_{19}$ ($x = 0$ wt%) sample surface under applied test loads (F) equal to 2.98 N and 9.80 N. At least three clear indentations were considered for each specific load. The average of the two diagonals (d) was calculated and Vickers microhardness (H_v) for $\text{Co}_x\text{BaFe}_{12}\text{O}_{19}$ samples was estimated with reference to the given equation [46]:

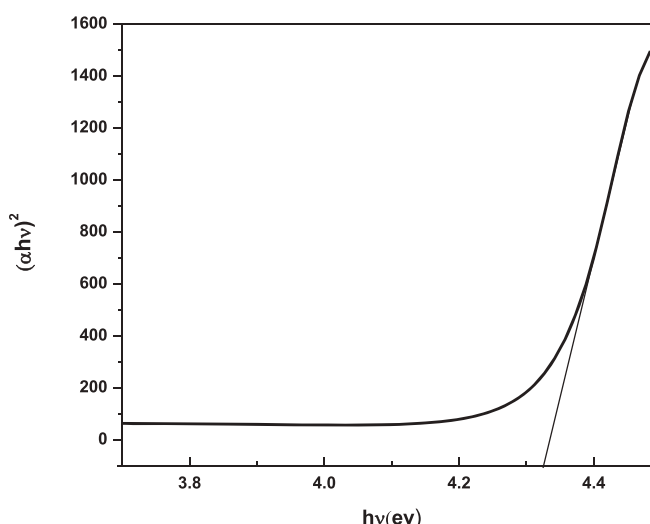


Figure 5. The plot of $(\alpha h\nu)^2$ versus photon energy $h\nu$ for $\text{Co}_x\text{BaFe}_{12}\text{O}_{19}$ nanoparticles with $x = 0$ wt% calcined at $T = 850^\circ\text{C}$.

Table 2. The optical band gap E_g for $\text{Co}_x\text{BaFe}_{12}\text{O}_{19}$ with $x = 0, 0.04, 0.06$ and 0.1 wt% at different calcination temperatures ($T = 850^\circ\text{C}, 900^\circ\text{C}, 950^\circ\text{C}$ and 1050°C).

x (wt%)	E_g (eV)			
	$T = 850^\circ\text{C}$	$T = 900^\circ\text{C}$	$T = 950^\circ\text{C}$	$T = 1050^\circ\text{C}$
0	4.25	4.05	3.83	3.71
0.04	4.05	3.83	3.75	—
0.06	4.00	3.81	3.71	—
0.1	3.95	3.66	3.65	3.57

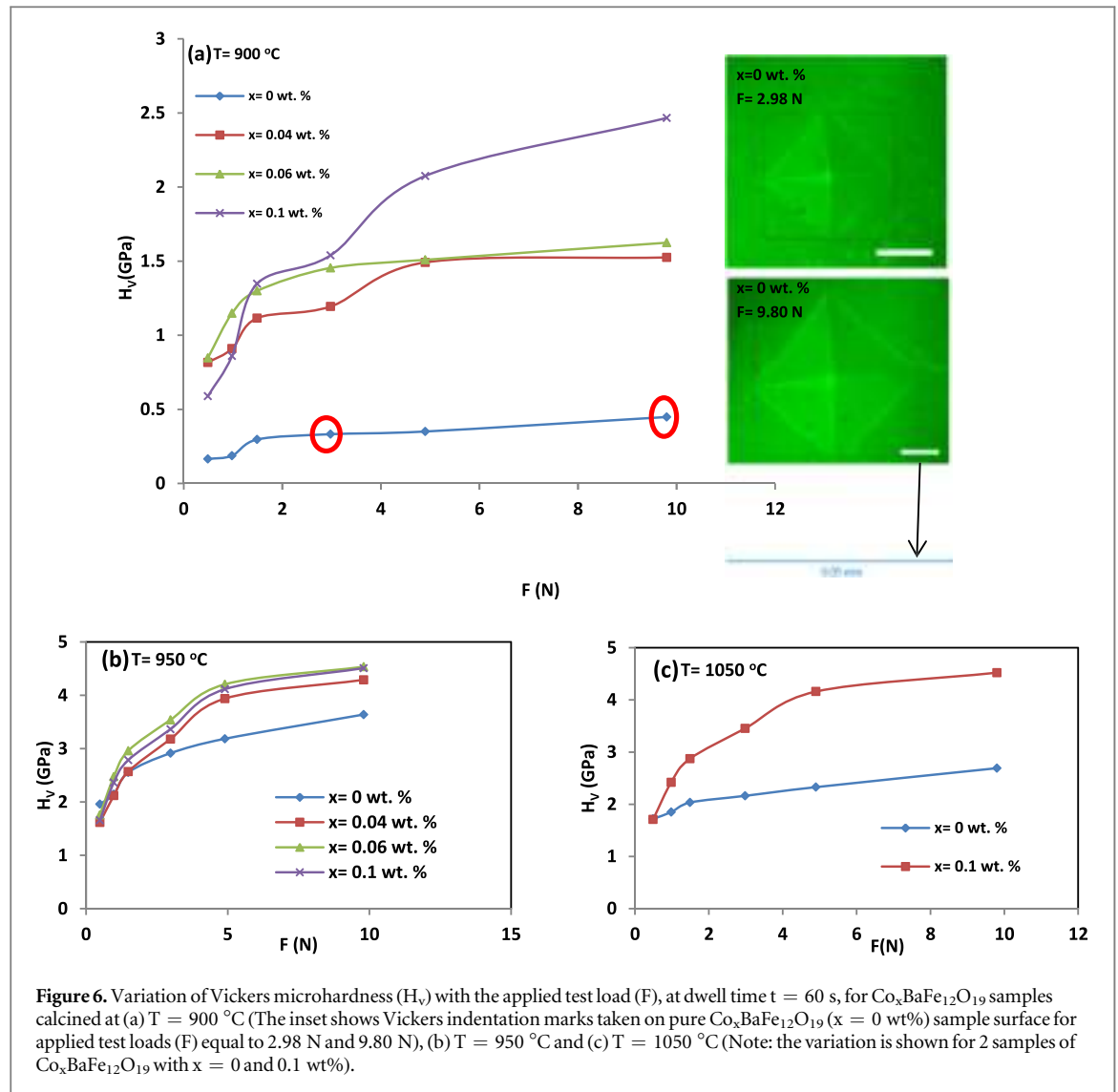
$$H_v = 1854.4 \left(\frac{F}{d^2} \right) (\text{GPa}), \quad (5)$$

where F is the applied indentation test load in Newton and d is the average diagonal length of indentation in μm .

The dependence of Vickers microhardness (H_v) on the applied indentation test load is known as indentation size effect (ISE). Usually two types of ISE could be observed for ceramics materials: (I) normal ISE (NISE) which involves a decrease in the apparent microhardness with increasing the applied test load and (II) reverse indentation size effect (RISE) where the apparent microhardness increases with increasing the applied test load. It can be seen, from figures 6(a)–(c), that Vickers microhardness H_v for all the prepared samples increases with increasing the applied test load F . Therefore, the samples undergo reverse indentation size effect (RISE).

Figure 7(a) displays the variation of H_v with Co addition for $\text{Co}_x\text{BaFe}_{12}\text{O}_{19}$ samples calcined at 900°C , 950°C and 1050°C under applied test load $F = 4.90$ N. It is clear, from figure 7(a), that the increase in the Co-addition (x) enhances the microhardness (H_v) values of the prepared samples at all calcination temperatures. Therefore, Cobalt addition has the ability to increase grain connectivity, reduce pores, enhance relative volume fraction of $\text{BaFe}_{12}\text{O}_{19}$ and to reduce resistance to crack propagation among grains. This result is consistent with XRD results. Figure 7(b) shows the change of H_v with applied load F for pure $\text{Ba}_{12}\text{Fe}_{19}\text{O}$ sample calcined at 900°C , 950°C and 1050°C . It is obvious that the H_v values of the pure sample increase with increasing the calcination temperature from 900°C to 1050°C . This enhancement in the microhardness values could be attributed to the increase of crystallinity and phase formation of $\text{BaFe}_{12}\text{O}_{19}$ phase in accordance to the reduction of Fe_2O_3 phase formation with the increase of calcination temperature. This result is consistent with XRD results that showed that samples calcined at 1050°C have nearly single phase of $\text{BaFe}_{12}\text{O}_{19}$ with low traces of Fe_2O_3 phase. Setiadi *et al* [29] found an increase in Vickers microhardness with increasing sintering temperature from 1000°C to 1100°C for $\text{BaFe}_{12}\text{O}_{19}$ added with Al_2O_3 -MnO. Also, similar results were reported by Rusianto *et al* [26] for $\text{Sr}_x\text{Ba}_{1-x}\text{Fe}_{12}\text{O}_{19}$ with $x = 0.00, 0.25, 0.50, 0.75$, and 1.00 sintered at various temperatures of 1000 , 1100 , and 1200°C .

The elastic modulus (E) and yield strength (Y) are related to Vickers microhardness by the relations [46]; $E = 81.9635 H_v$ and $Y = \frac{H_v}{3}$, respectively. figures 8(a) and (b) shows the variation of elastic modulus (E) and



yield strength (Y) as function of Co-addition for $\text{Co}_x\text{BaFe}_{12}\text{O}_{19}$ calcined at 900 °C, 950 °C and 1050 °C (Note: E and Y are calculated from an average value for H_v taken at the last applied test loads $F = 2.98, 4.90$ and 9.80 N, plateau region). Figure 8(a) and b clarifies that E and Y increase as the Co-content increases. Therefore, they follow the same H_v -Co % variation trend. Zewu *et al* [47] examine the relation of elastic modulus to determine the critical grain size above which the micro-cracking will initiate due to anisotropic thermal stresses that appear during processing. The greater elastic modulus (E) and yield strength (Y) of a sample, the stronger connection between atoms or molecules in materials.

3.5.1. Indentation-induced cracking model (IIC)

In this model, Li and Bradt [48] try to explain the reverse ISE by considering that at maximum penetration, the applied indentation test load is balanced by the total specimen resistance which results from the following four factors: (1) friction at the indenter/specimen facet interface (frictional component), (2) elastic deformation, (3) plastic deformation and (4) specimen cracking. According to this model the indentation cracking is responsible for the reverse ISE, while frictional and elastic effects give rise to the normal ISE. In the case of indentation cracking, the Vickers microhardness measured is given by [49]

$$H_v = \lambda_1 k_1 \left(\frac{F}{d^2} \right) + k_2 \left(\frac{F^{5/3}}{d^3} \right), \quad (6)$$

where d is the indentation diagonal length, and λ_1 , k_1 and k_2 are constants. The constant k_2 depends on the applied load F , while k_1 is a constant depends on the indenter geometry. For an ideally perfect plastic body $H_v = k_1 \left(\frac{F}{d^2} \right)$ here $\lambda_1 = 1$ and $k_2 \left(\frac{F^{5/3}}{d^3} \right) = 0$. In the case of a perfect brittle solid, $H_v = k_2 \left(\frac{F^{5/3}}{d^3} \right)$ and $\lambda_1 = 0$. For samples showing reverse ISE the microhardness may be represented by the empirical relation [45]

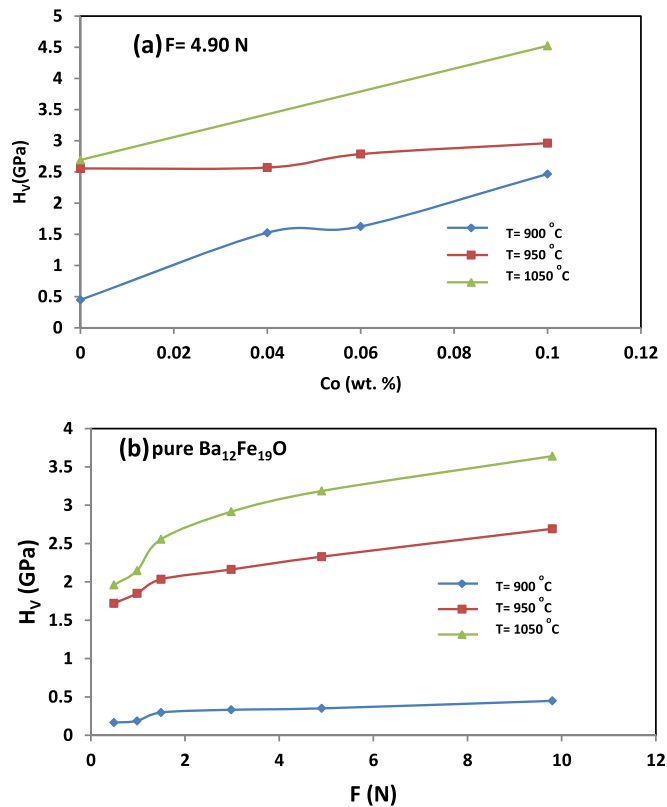


Figure 7. (a) The variation of H_v with Co addition for $\text{Co}_x\text{BaFe}_{12}\text{O}_{19}$ samples calcined at 900 °C, 950 °C and 1050 °C under applied test load $F = 4.90$ N (Note: the variation at 1050 °C is shown for 2 samples of $\text{Co}_x\text{BaFe}_{12}\text{O}_{19}$ with $x = 0$ and 0.1 wt%). (b) The change of H_v with applied load F for pure $\text{Ba}_{12}\text{Fe}_{19}\text{O}$ sample calcined at 900 °C, 950 °C and 1050 °C.

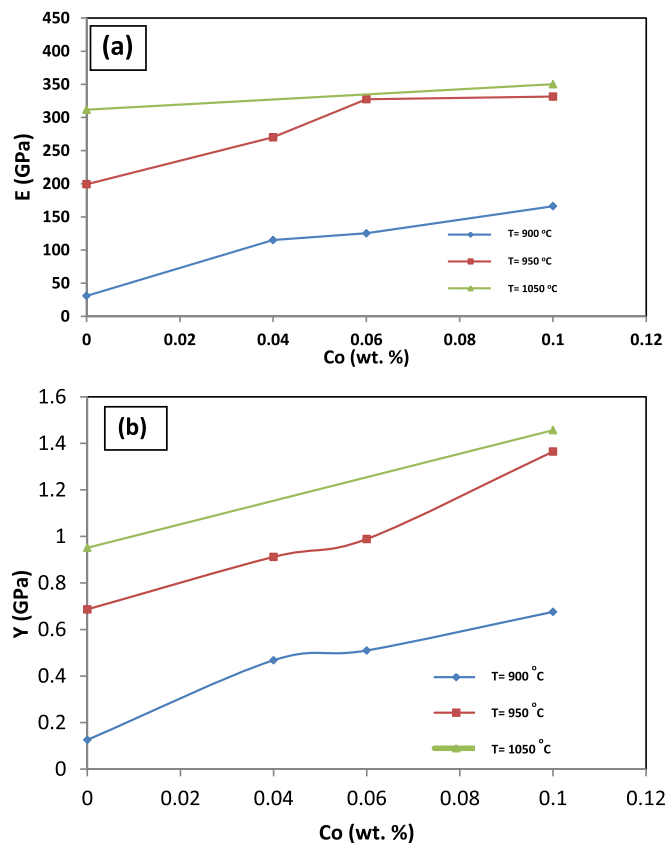


Figure 8. (a) the variation of elastic modulus (E) and (b) yield strength (Y) as function of Co-addition for $\text{Co}_x\text{BaFe}_{12}\text{O}_{19}$ calcined at 900 °C, 950 °C and 1050 °C (Note: the variation at 1050 °C is shown for 2 samples of $\text{Co}_x\text{BaFe}_{12}\text{O}_{19}$ with $x = 0$ and 0.1 wt%).

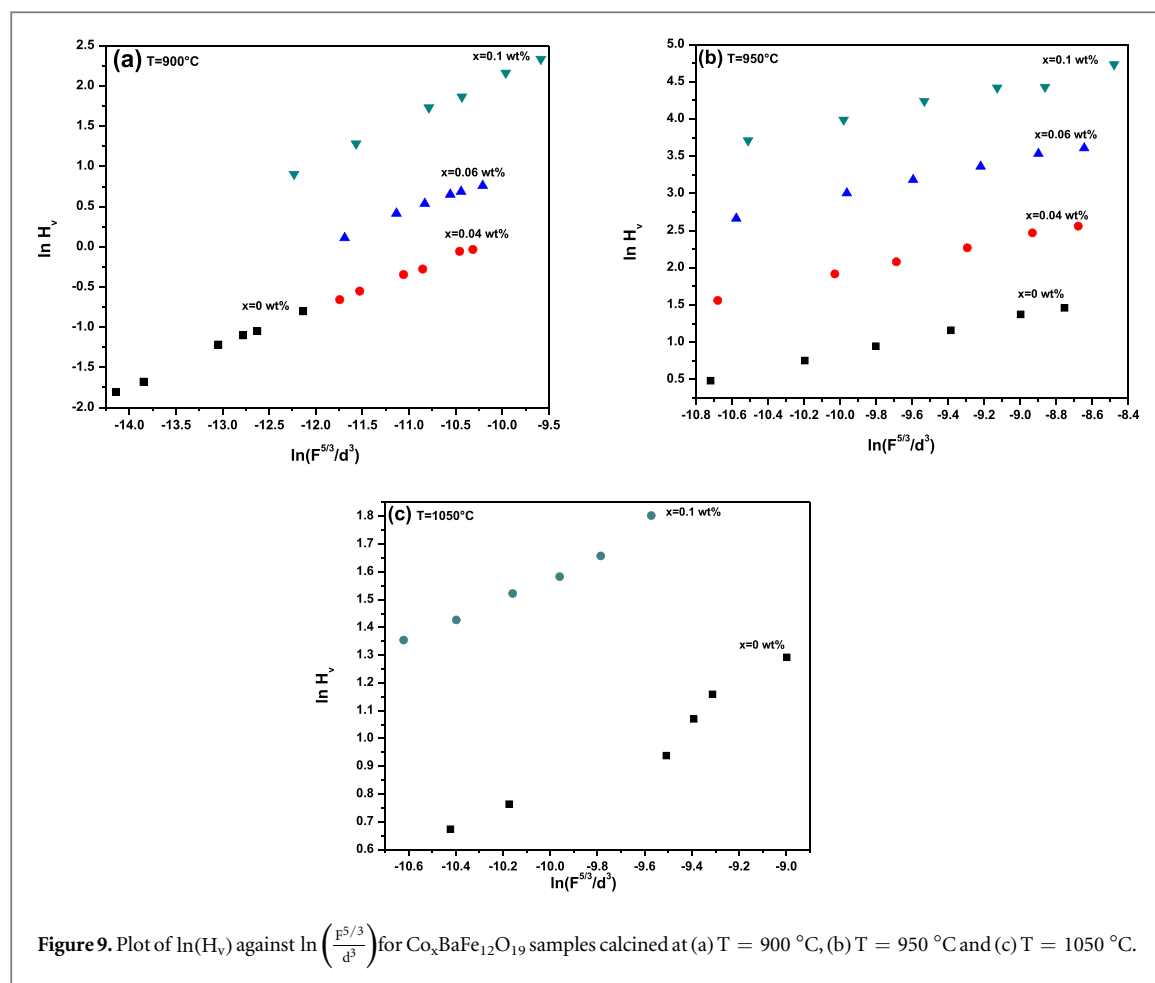


Table 3. The fitting parameters (k and m) obtained from indentation induced cracking model, experimental and theoretical load independent microhardness for $\text{Co}_x\text{BaFe}_{12}\text{O}_{19}$ samples calcined at $T = 900^\circ\text{C}$, 950°C and 1050°C .

	x (wt%)	IIC Model			
		k ($\text{N}^{(3-5m)/3}/\mu\text{m}^{(2-3m)}$)	m	$(H_v)_{\text{exp.}}$ (GPa)	H_{IIC} (GPa)
Calcined at 900°C	0	214	0.50	0.40	0.40
	0.04	151	0.44	1.51	1.49
	0.06	149	0.44	1.57	1.54
	0.1	450	0.45	2.27	2.27
Calcined at 950°C	0	358	0.50	4.11	4.11
	0.04	361	0.50	4.31	4.26
	0.06	340	0.49	4.37	4.40
	0.1	290	0.47	4.47	4.48
Calcined at 1050°C	0	141.4	0.412	3.41	3.42
	0.04	—	—	—	—
	0.06	—	—	—	—
	0.1	131.47	0.41	2.51	2.52

$$H_v = k \left(\frac{F^{5/3}}{d^3} \right)^m, \quad (7)$$

where k and the exponent m are load independent constants. The indentation size effect behavior can be identified from the value of m such that, $m > 0.6$ refers to normal ISE while $m < 0.6$ refers to RISE [50].

Figures 9(a)–(c) illustrates the plot of $\ln(H_v)$ against $\ln\left(\frac{F^{5/3}}{d^3}\right)$ for $\text{Co}_x\text{BaFe}_{12}\text{O}_{19}$ samples calcined at 900°C , 950°C and 1050°C , respectively. The values of m and k are calculated from the plotting graphs using equation (7) and their values are listed in table 3. It is shown, from table 3, that values of m are less than 0.6 for all samples indicating the reverse ISE.

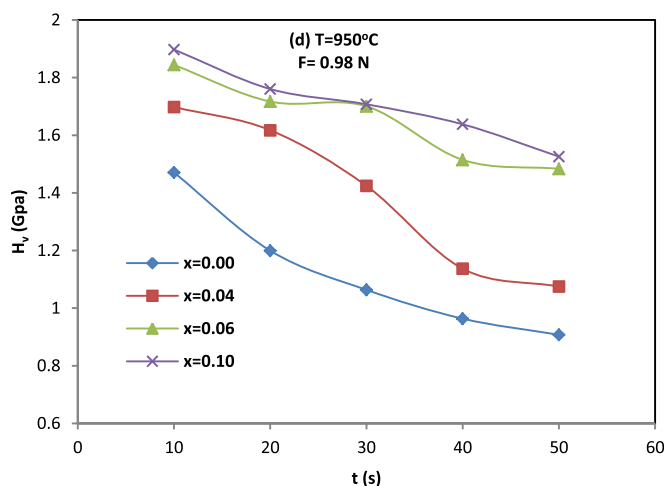


Figure 10. Variation of H_v with the dwell time (t) for $\text{Co}_x\text{BaFe}_{12}\text{O}_{19}$ with $x = 0, 0.04, 0.06$ and 0.1 wt% calcined at $T = 950$ °C and with fixed load $F = 0.98$ N.

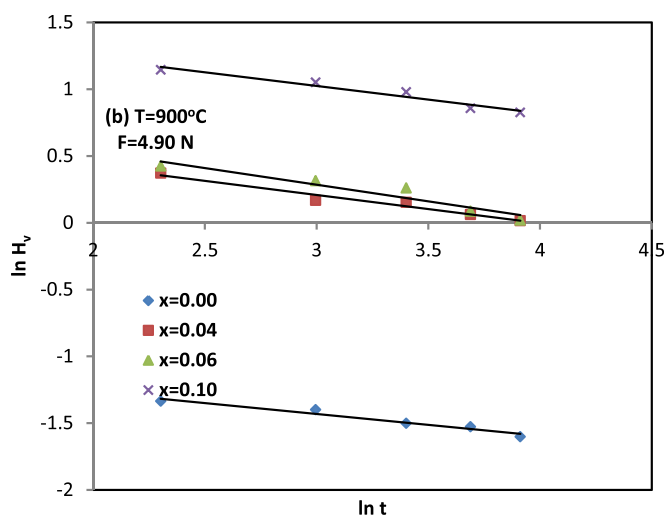


Figure 11. The fitted data according to Sargent- Ashby model for $\text{Co}_x\text{BaFe}_{12}\text{O}_{19}$ with $x = 0, 0.04, 0.06$ and 0.1 wt% at calcination temperature $T = 900$ °C and fixed load $F = 4.90$ N.

The theoretical microhardness values according to the indentation induced cracking model H_{IIC} were calculated as a function of the applied test load F according to equation (7) and their values are listed in table 3 together with the experimental microhardness values $(H_v)_{exp.}$ estimated in the plateau region. It is clear that the theoretical data, calculated according to IIC model, is well matched the experimental measured data. Therefore, it can be concluded that the indentation induced cracked model IIC is a suitable model that fits our experimental microhardness data and explains the reverse indentation size effect (RISE) nature of the prepared samples.

3.5.2. Time dependent Vickers microhardness (Indentation creep)

Time dependent movement of a hard indenter into a solid, under fixed applied load, is known as indentation creep. The measurements of indentation creep can be seen as a fast, clear and anti-destructive technique for gathering information on the mechanical performance of materials [51]. Indentation creep test was performed on our samples to examine the power law indentation creep performance of Cobalt added Barium hexaferrite nanoparticles calcined at 900 °C, 950 °C and 1050 °C.

Figure 10 displays the variation of H_v with the dwell time (t) at fixed applied load $F = 0.98$ N, for $\text{Co}_x\text{BaFe}_{12}\text{O}_{19}$ with $x = 0.00, 0.04, 0.06$ and 0.10 wt% calcined at 950 °C. It is evident from the figure that with rising dwell time, microhardness declines and there are two stages of transitions. The first stage is characterized by a rapid reduction in H_v - t curves for the duration of a dwell time $t < 40$ s, followed by a second stage that

Table 4. Stress exponents for $\text{Co}_x\text{BaFe}_{12}\text{O}_{19}$ with $x = 0.00, 0.04, 0.06$ and 0.10 wt% calcined at 900°C and 950°C at constant applied loads $F = 0.98, 4.90$ and 9.80 N.

x (wt%)	T = 900°C		
	M		
	F = 0.98 N	F = 4.90 N	F = 9.80 N
0.00	6.16	6.16	3.17
0.04	4.09	4.73	3.62
0.06	4.55	4.11	5.99
0.10	4.09	4.88	7.74

x (wt%)	T = 950°C		
	M		
	F = 0.98 N	F = 4.90 N	F = 9.80 N
0.00	3.30	5.94	3.70
0.04	3.35	3.29	3.41
0.06	7.38	5.39	3.64
0.10	8.07	5.80	2.93

occurs at dwell-time t greater than 40 s where microhardness is gradually reduced at a low rate as the dwell-time increases. The H_V - t trend, demonstrating the nature of the indenter's penetration depth and illustrates the typical behavior between H_V and t [52]. This is also a predictor of the material bearing creep deformation [53].

Sargent- Ashby model [54] can be used to test the indentation creep nature of the prepared samples. The time dependent microhardness is calculated by the following relationship, according to this model;

$$H_V(t) = \frac{\varepsilon_0}{(nc\varepsilon_0 t)^{\frac{1}{\mu}}}, \quad (8)$$

where ε_0 is the strain rate at reference stress r_0 , c is a constant and μ is the stress exponent.

Figure 11 demonstrates the experimental data fitted to Sargent-Ashby model for $\text{Co}_x\text{BaFe}_{12}\text{O}_{19}$ with $x = 0.00, 0.04, 0.06$ and 0.10 wt% calcined at 900°C at constant load $F = 4.90$ N. Straight lines with slopes equal to the negative reverse stress exponent $-1/\mu$ are obtained from $\ln(H_V)$ versus $\ln(t)$ plot. The fitted lines can be seen nearly parallel. The parallel lines reveal that cobalt additions do not affect the stress exponents significantly [55]. The calculated stress exponent values for the applied test loads ($F = 0.98, 4.90$ and 9.80 N) for samples calcined at 900°C and 950°C are listed in table 4. It is clear, from table 4, that the stress exponents μ for all samples calcined at 900°C have values that range from 3.1 to 7.7 while samples calcined at 950°C have the stress exponents μ ranges from 2.9 to 8.0 . In addition, with respect to the power law creep; the higher the stress exponent, the higher the yield strength [56, 57]. The specimens are therefore more resistant to indentation creeps for greater loads. Several details on the deformation mechanism can be shown from the value of the stress exponent μ . When μ has a value nearly one, it means a diffusion creep in the sample [58], a grain boundary sliding [55, 56] will occur at a value of μ around two. Dislocations climbs arise for μ values between 4 and 6 [59]. Dislocation creep, however, is governed when μ has values between 3 and 10 [60, 61]. The numerical values of μ range from 3.1 to 8.0 for the prepared samples. Therefore, the samples investigated exhibit grain boundary sliding, accompanied by dislocation climbs at small loads followed by dislocation creep for great loads throughout the operative creep mechanism.

4. Conclusion

Chemical co-precipitation method is successfully utilized to prepare nano-scale particles of pure Barium hexaferrite $\text{BaFe}_{12}\text{O}_{19}$ and Cobalt added Barium hexaferrite $\text{Co}_x\text{BaFe}_{12}\text{O}_{19}$, with $x = 0.04, 0.06$ and 0.1 wt%. The effect of Cobalt addition and calcination temperature on the microstructure, morphology, optical and mechanical properties of the synthesized samples was investigated. XRD results revealed the hexagonal structure of the prepared samples and indicated enhancement in the phase formation of $\text{BaFe}_{12}\text{O}_{19}$ and reduction of Fe_2O_3 phase with increasing the calcination temperature. Also, it was concluded from XRD that the optimum calcination temperature for nearly single phase of $\text{BaFe}_{12}\text{O}_{19}$ is at $T = 1050^\circ\text{C}$. The lattice parameters (a and c) and unit cell volume (V) were almost unchanged with either Cobalt addition or calcination temperatures. By the use of Debye-Scherrer equation, the crystallite size was calculated to be in the range of 51 – 142 nm. FTIR spectra

confirmed the formation of hexaferrite phase through the existence of strong absorption peaks that appeared between 581 cm^{-1} and 435 cm^{-1} . TEM micrographs clarified that the samples composed of agglomerated grains that form clusters of different sizes and shapes. Moreover, the hexagonal shaped particles become clearly defined as the Cobalt content and calcination temperature were increased to 0.1 wt% and 1050°C , respectively. An absorbance peak was observed from the UV- visible spectroscopy at wavelength of 352 nm for $\text{Co}_x\text{BaFe}_{12}\text{O}_{19}$ with $x = 0.1\text{ wt\%}$ was slightly shifted to lower wavelengths with increasing the calcination temperature. It was concluded that the absorbance shift depends mainly on various factors such as band gap, grain size, impurities, oxygen deficiency and surface effects. Moreover, the optical band gap E_g for the samples was estimated from Tauc relation as function of both calcination temperature (T) and Cobalt addition (x). It was found that both Co addition and calcination temperatures decrease the band gap of barium hexaferrite nanoparticles. This reduction was attributed to the enhancement in the samples grain size which is consistent with TEM results. Vickers microhardness was employed to investigate the mechanical performance of the prepared samples. Microhardness results revealed that the samples experience reverse indentation size effect (RISE) attitude. It was concluded that Vickers microhardness, elastic modulus and yield strength of the prepared samples were enhanced with increasing Co-addition and calcination temperature. Thus, Cobalt in $\text{Ba}_{12}\text{Fe}_{19}\text{O}$ matrix and elevated calcination temperatures have the ability to enhance the mechanical properties and microstructure of the prepared samples. In addition, indentation induced cracked model (IIC) was found to be an adequate model for the description of the microhardness data. Indentation creep results showed that the samples investigated exhibit grain boundary sliding, accompanied by dislocation climbs at small loads followed by dislocation creep for great loads throughout the operative creep mechanism.

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