

# Effects of Substrate Temperature, Oxygen Pressure and Laser Fluence on Structural and Magnetic Properties of Pulsed Laser-Deposited Cobalt Ferrite Thin Films

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**Abstract** Cobalt ferrite (CFO) thin films were grown on fused quartz substrate using pulsed laser deposition technique. The effects of various process parameters viz. substrate temperature, oxygen pressure, and laser energy fluence on structural, morphological, and magnetic properties were studied using X-ray diffraction (XRD), atomic force microscope (AFM), and vibrating sample magnetometer (VSM), respectively. The film deposited at the lower substrate temperature of 400 °C showed growth of CFO along preferred [110] direction and becomes textured for the substrate temperature of 800 °C with increase in grain size from 21 to 46 nm measured using AFM. The value of saturation magnetization ( $4\pi M_s$ ) increases from 1940 to 2460 G with increase in substrate temperature from 400 to 800 °C, respectively. The CFO thin film deposited at 400 °C showed a coercivity value of 1880 Oe, which decreased to 670 Oe for 800 °C. The  $4\pi M_s$  values first decrease with increase in oxygen pressure up to 0.4 mbar then increased for higher oxygen pressures. The  $H_c$  value increases from 670 to 1290 Oe when oxygen pressure is increased from 0.2 to 0.4 mbar, which finally decreased to 1100 Oe for an oxygen pressure of 0.8 mbar. The structural and magnetic properties of CFO thin films are also observed to be influenced greatly by laser energy fluence. The varied magnetic properties of CFO

thin films at different deposition conditions in pulsed laser deposition are interesting and may be useful for possible applications.

**Keywords** Cobalt ferrite · X-ray diffraction · Magnetization · Thin film · Pulsed laser deposition

## 1 Introduction

Thin films of spinel ferrites have been studied for their excellent electric and magnetic properties. Among these spinel ferrites, cobalt ferrite ( $\text{CoFe}_2\text{O}_4$ ) thin films have proven their candidature for numerous applications in magnetic recording, microelectro-mechanical system devices, etc. due to its high Curie temperature, high saturation magnetization, and high coercivity [1–6]. The cobalt ferrite follows an inverse spinel structure with a general formula for the ion distribution  $\text{A}^{3+}[\text{B}^{2+}\text{B}^{3+}]\text{O}_4^{2-}$ . In this structure, half of  $\text{Fe}^{3+}$  cations are occupied in tetrahedral A sites, while the remaining half of the  $\text{Fe}^{3+}$  cations along with divalent  $\text{Co}^{2+}$  cations are occupied in octahedral B lattice sites. Co-occurrence of  $\text{Fe}^{3+}$  and  $\text{Co}^{2+}$  cations in the same B (octahedral) sites lead to magnetization in this ferrite.

There had been different methods, namely pulsed laser deposition (PLD), sputtering, sol–gel method, spray pyrolysis, and molecular beam epitaxy, for the growth of cobalt ferrite thin films [7–11]. Among these methods, PLD has established its importance due to epitaxial growth in thin films besides maintaining the same stoichiometry in thin films as that of the target material [12–17].

Thin films of  $\text{CoFe}_2\text{O}_4$  have been reported to grow at various in situ substrate temperatures and oxygen partial pressure in recent past [15, 16]. These films showed preferred orientation along [111] when deposited on Si(100)

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substrate at various temperatures up to 700 °C and pressures up to 10 Pa. The films have been observed to possess high coercivity ( $\sim 2000$  Oe). The reduction of oxygen vacancies at higher oxygen pressure resulted into the observed values of coercivity. In addition, there had been monotonic increase in magnetization with pressure but with the substrate temperature magnetization first increases until 600 °C then decreases.

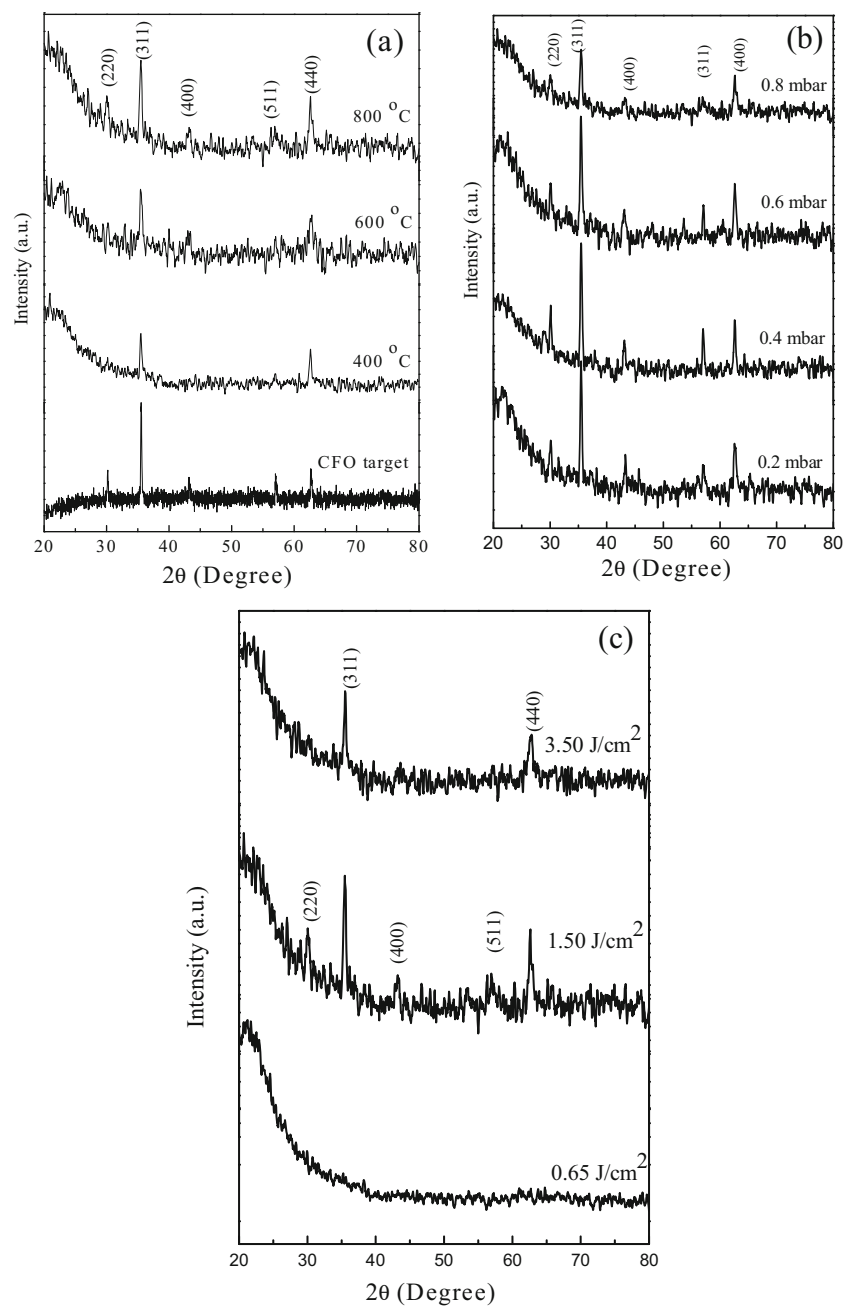
In this paper, we present a study on the influence of substrate temperature, oxygen pressure, and energy fluence on the evolution of structural and magnetic properties of

cobalt ferrite thin films deposited by PLD on fused quartz substrates.

## 2 Experimental Details

PLD was used for the deposition of cobalt ferrite (CFO) thin films on quartz substrate using single-phase high-quality CFO target. The CFO target used for the deposition was prepared by conventional solid-state reaction method. For the synthesis of CFO target, the constituent oxides ( $\text{Co}_3\text{O}_4$  and

**Fig. 1** XRD pattern of  $\text{CoFe}_2\text{O}_4$  thin films deposited at **a** different substrate temperatures (XRD of bulk CFO is also shown for comparison), **b** different oxygen pressures, and **c** different energy fluences



**Table 1** Relative intensities of different (*hkl*) planes of CoFe<sub>2</sub>O<sub>4</sub> ICDD (file no. 22-1086), target, and thin films deposited at different substrate temperatures (*T<sub>s</sub>*)

<i>hkl</i>	Relative intensity (%)		<i>T<sub>s</sub></i>		
	ICDD	Bulk (target)	400 °C	600 °C	800 °C
(220)	35.53	30.72	–	–	32.87
(311)	100.00	100.00	100.00	100.00	100.00
(400)	14.91	12.86	–	55.85	28.69
(511)	31.13	31.03	–	–	22.03
(440)	36.73	36.18	69.94	66.17	36.42

Fe<sub>2</sub>O<sub>3</sub>) with 99.99 % purity (Sigma Aldrich) were weighed in stoichiometric ratio and mixed thoroughly using acetone as a grinding medium. The target was pressed into a disc sample of 20-mm diameter and sintered at 600 °C for 12 h. Again, it was grinded and pressed into a disc sample and sintered at 800 °C for 12 h and finally sintered at 1000 °C for 4 h. The quartz substrates used for the deposition of CFO thin films were cleaned prior to the deposition. For cleaning of the substrate, firstly, it was washed several times with distilled water, then placed in the solution of ammonium hydroxide (NH<sub>4</sub>OH), hydrogen peroxide, and deionized water in the ratio 1:1:2 heated at 80 °C. The substrates were then washed with distilled water followed by degreasing with acetone and isopropanol. In PLD, the second harmonic (532 nm) of Nd:YAG laser with 10 Hz repetition rate and 5–6 ns pulse width was used for the deposition of CFO thin films. The base vacuum of 2.0 × 10<sup>-6</sup> mbar was maintained in the chamber prior to deposition. The films were deposited in three series, A—at different substrate temperatures (400, 600, and 800 °C), 0.2 mbar of oxygen pressure, and 1.5 J/cm<sup>2</sup> energy fluence; B—at various oxygen pressures (0.2, 0.4, 0.6, and 0.8 mbar), 800 °C substrate temperature, and 1.5 J/cm<sup>2</sup> energy fluence; and C—at various laser energy fluence (0.65, 1.5, and 3.5 J/cm<sup>2</sup>), 800 °C substrate temperature, and 0.2 mbar of oxygen pressure. The deposition time and the target to substrate

distance were kept fixed to 20 min and 4.5 cm, respectively, for all the films.

The structural properties of the films were studied using X-ray diffraction (XRD) in 2θ ranging from 20° to 80° (Rigaku SmartLab diffractometer, λ<sub>CuKα</sub> = 1.5405 Å). The morphology of the films was analyzed by atomic force microscopy (AFM) (Agilent 5500 scanning probe microscope). The magnetic measurements (applied magnetic field (*H*) vs magnetization (*M*), *M*–*H* loops) were carried out using VersaLab (Quantum Design) vibrating sample magnetometer (VSM) in parallel configuration (in-plane), i.e., *H* || to film plane at room temperature. The diamagnetic contribution of fused quartz substrate was corrected to obtain *M*–*H* loops. Thicknesses of the films were measured using spectroscopic ellipsometer (J. A. Woollam) and were around 100 nm.

### 3 Result and Discussion

The XRD of CFO thin films deposited with varying deposition parameters are shown in Fig. 1a–c. Figure 1a shows the XRD of CFO thin films deposited at different substrate temperatures of 400, 600, and 800 °C (A series) (XRD of bulk CFO target is also shown for comparison). The films deposited at all substrate temperatures were indexed to

**Table 2** Relative intensities of different (*hkl*) planes of CoFe<sub>2</sub>O<sub>4</sub> ICDD (file no. 22-1086), target, and thin films deposited at various oxygen pressures (*P<sub>O2</sub>*)

<i>hkl</i>	Relative intensity (%)		<i>P<sub>O2</sub></i>			
	ICDD	Bulk (target)	0.2 mbar	0.4 mbar	0.6 mbar	0.8 mbar
(220)	35.53	30.72	32.87	39.32	33.89	40.57
(311)	100.00	100.00	100.00	100.00	100.00	100.00
(400)	14.91	12.86	28.69	24.00	32.22	26.68
(511)	31.13	31.03	22.03	37.34	27.75	24.62
(440)	36.73	36.18	36.42	43.44	45.40	68.82

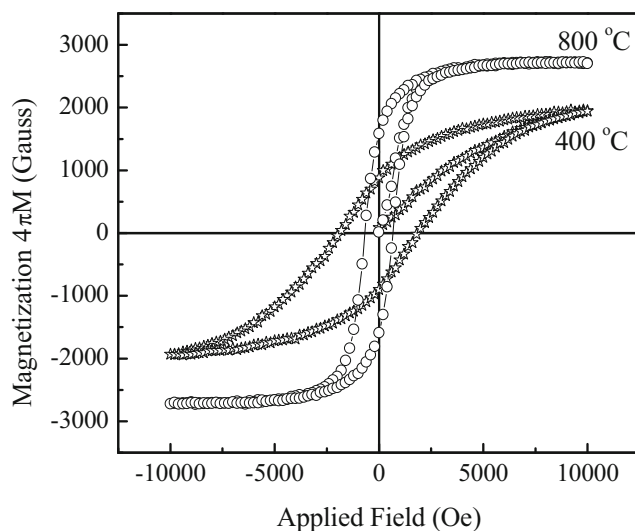
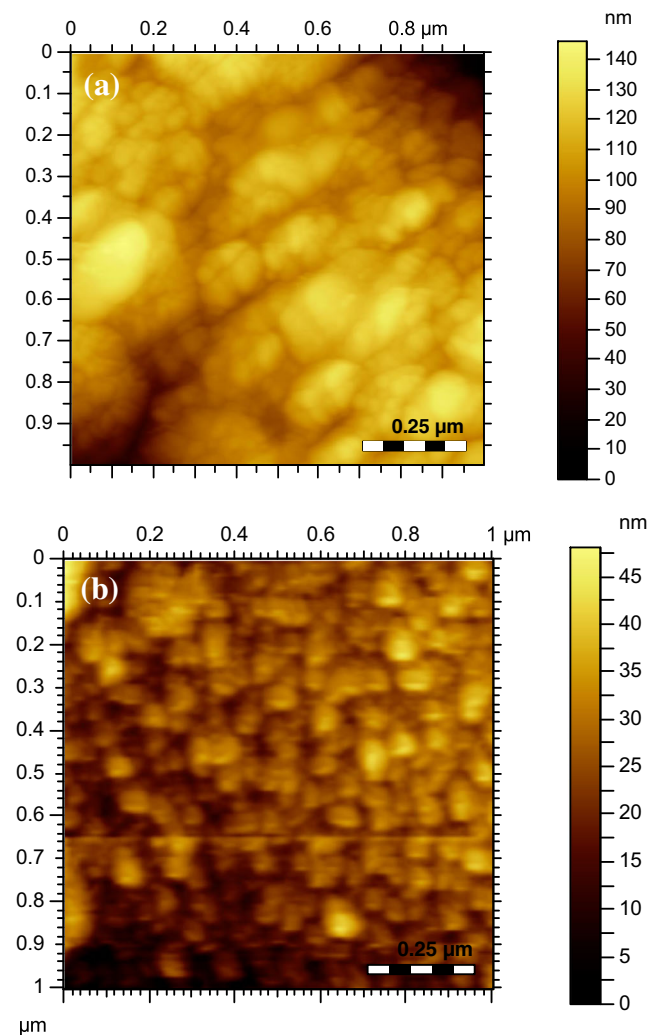
**Table 3** Relative intensities of different (*hkl*) planes of CoFe<sub>2</sub>O<sub>4</sub> ICDD (file no. 22-1086), target, and thin films deposited at different laser energy fluences

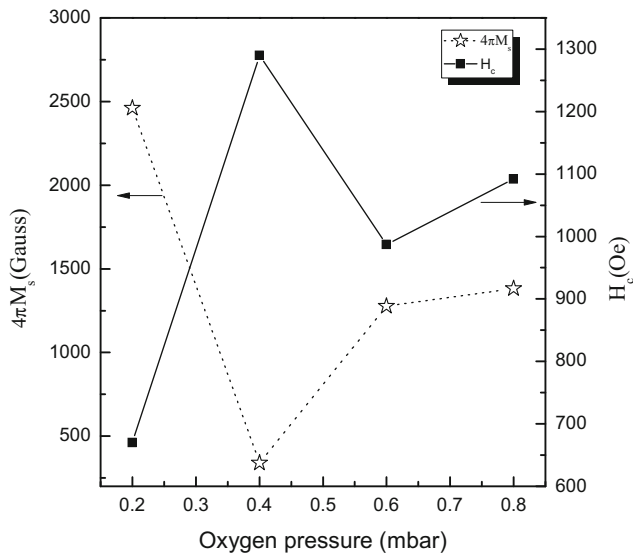
<i>hkl</i>	Relative intensity (%)		Energy fluence		
	ICDD	Bulk (target)	0.65 J/cm <sup>2</sup>	1.5 J/cm <sup>2</sup>	3.5 J/cm <sup>2</sup>
(220)	35.53	30.72	–	32.87	–
(311)	100.00	100.00	–	100.00	100.00
(400)	14.91	12.86	–	28.69	–
(511)	31.13	31.03	–	22.03	–
(440)	36.73	36.18	–	36.42	50.25

sing-phase CFO (International Centre for Diffraction Data (ICDD) file no. 22-1086). It is interesting to note that the film deposited at 400 °C shows only two peaks of (311) and (440) reflections following relative intensities of its bulk counterpart (details of relative intensities of different *hkl* planes of CFO films deposited at different temperatures are tabulated in Table 1). It is worthy to note that the XRD of the film deposited at 400 °C was devoid of next higher intensity peaks (220) and (511) as observed in bulk. Film deposited at 600 °C showed an additional XRD peak corresponding to (400) reflection, while a maximum of five XRD peaks were observed for the film deposited at 800 °C. The relative intensities of the peaks other than the maximum intensity peak (311) for the films deposited at 600 and 800 °C do not exactly follow the bulk behavior suggesting texture growth of CFO at elevated substrate temperatures, while preferred growth along [440] direction for lower substrate temperature of 400 °C. CFO thin films, when deposited at varying oxygen pressures (*P*<sub>O<sub>2</sub></sub>), show all XRD peaks as observed in bulk (Fig. 1b) with slight variation in their intensities (Table 2), while the films deposited at the highest energy

fluence of 3.5 J/cm<sup>2</sup> show the growth of CFO with [440] as the preferred growth direction (Fig. 1c and Table 3).

The growth of cobalt ferrite thin films with (111)-preferred orientation has been reported when deposited at substrate temperatures  $\geq$  above 650 °C on Si(100) substrates by Zhou et al. [15]. On the other hand, films

**Fig. 2** Magnetic hysteresis loops (*M–H*) of CoFe<sub>2</sub>O<sub>4</sub> thin film deposited at 400 and 800 °C substrate temperatures**Fig. 3** AFM images of CoFe<sub>2</sub>O<sub>4</sub> film grown at a 400 °C and b 800 °C



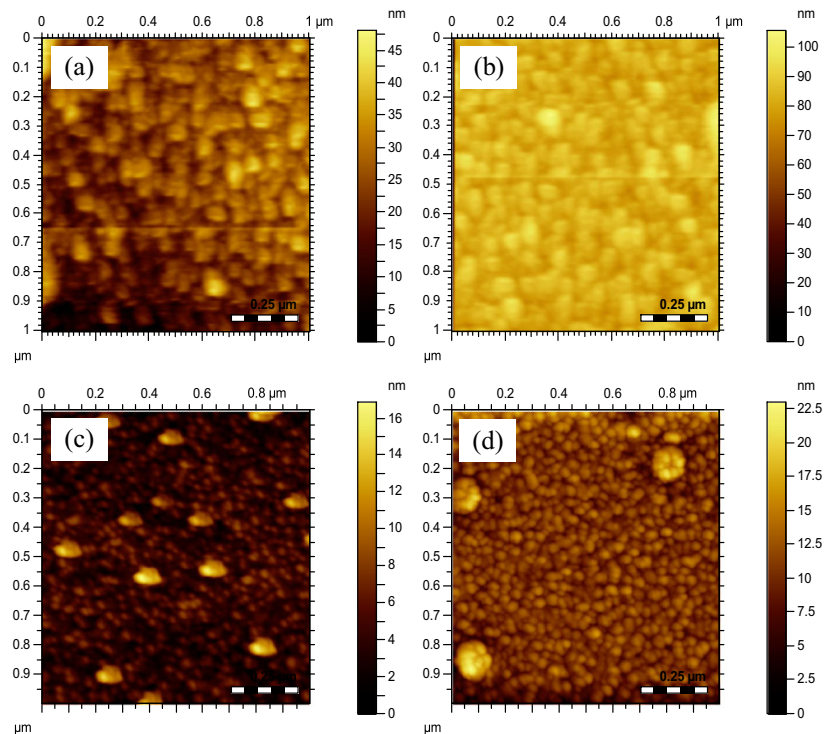
**Fig. 4** The values of  $4\pi M_s$  and  $H_c$  as a function of oxygen pressure ( $P_{O_2}$ ) measured from  $M-H$  loop of  $CoFe_2O_4$  thin films deposited at a substrate temperature of 800 °C. The values of  $4\pi M_s$  and  $H_c$  as a function of oxygen pressure ( $P_{O_2}$ ) measured from  $M-H$  loop of  $CoFe_2O_4$  thin films deposited at a substrate temperature of 800 °C

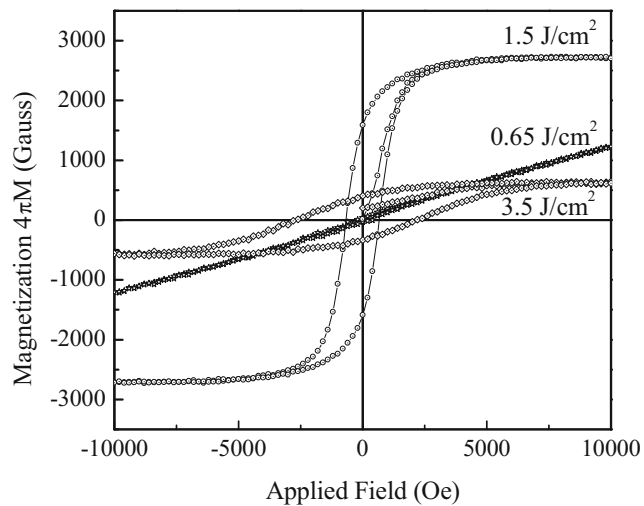
deposited at lower temperatures (350–600 °C) showed textured growth with (111), (311), and (400) reflections. The intensities of (311) and (400) peaks were reported to reduce with the increase in substrate temperature resulting preferential growth along (111) at elevated temperatures. Additionally, it has also been reported that oxygen pressure also

played an important role in film texture. Films deposited between 1 and 5 Pa of oxygen pressure were reported to be oriented along the (111) direction while films deposited at 10 Pa of pressure were disordered. The grains grown by higher laser ablation had enough mobility to find their proper position onto the thermal substrate. We also observed a similar trend in orientation of growth of CFO thin films along the (110) direction at lower oxygen pressure. Yin et al. [16] have reported the growth of CFO thin films with (220) textured orientation at substrate temperatures below 500 °C on quartz substrate and at higher temperatures film became highly (111) textured. The textured growth was suggested due to possible perpendicular anisotropy in CFO thin films at these temperatures.

The magnetic properties (magnetization versus applied magnetic field ( $M-H$  hysteresis loop)) of CFO thin films were measured at room temperature in parallel configuration, i.e., magnetic field was applied parallel to thin film’s plane at 300 K. Figure 2 shows the  $M-H$  loops of the films deposited at 400 and 800 °C (series A). It is interesting to note from these  $M-H$  loops that the loop shows its hardness to saturate up to a maximum of 10,000 Oe of applied field for the film deposited at 400 °C while the  $M-H$  loop for the film deposited at a higher substrate temperature of 800 °C saturates for the same applied field. The value of saturation magnetization ( $4\pi M_s$ ) was measured as intercept on y-axis ( $M$ -axis) by drawing tangential line from the high-field loops to the zero field. The value of  $4\pi M_s$  was found to increase from 1940 to 2460 G while

**Fig. 5** AFM images of  $CoFe_2O_4$  film grown at **a** 0.2 mbar, **b** 0.4 mbar, **c** 0.6 mbar, and **d** 0.8 mbar of oxygen pressure ( $P_{O_2}$ )





**Fig. 6**  $M$ – $H$  loops of the film deposited at an energy fluence of 0.65, 1.5, and 3.5  $\text{J}/\text{cm}^2$

coercivity ( $H_c$ ) decreased from 1880 to 670 Oe when substrate temperature is increased from 400 to 800 °C. The values of saturation magnetization for these films are lower than the bulk value of magnetization (5026 G). The lower value of magnetization as compared to the bulk has already been reported and suggested that it could be due to a large grain boundary volume present in thin films [19, 20]. Observation of increase in magnetization with the increase in substrate temperature may be understood due to growth of grains at elevated temperatures [20]. AFM images of the films showed the growth of grain size from 21 to 46 nm for the substrate temperatures of 400 and 800 °C, respectively (Fig. 3). Exhibition of hard magnetization for the film deposited at a substrate temperature of 400 °C may be explained due to the fact that easy axis of magnetization along [110] does not lie within the film plane and is in line with the XRD data of the film. However, textured growth of CFO film at a higher substrate temperature of 800 °C may result into magnetic anisotropy of the films. Suzuki et al. [18] has also reported hard axis of magnetization in highly [110]-oriented CFO thin films deposited onto  $\text{CoCr}_2\text{O}_4$  (CCO)-buffered  $\text{SrTiO}_3$  (STO) and  $\text{MgAl}_2\text{O}_4$  substrate.

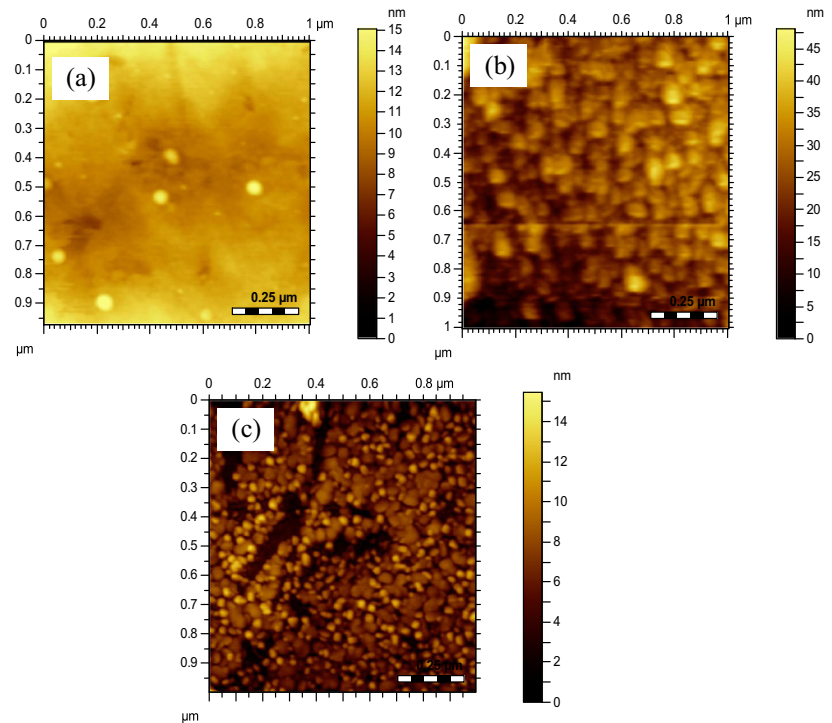
The values of  $4\pi M_s$  and  $H_c$  measured from  $M$ – $H$  loops (not shown) as a function of oxygen pressure for the CFO thin films deposited at a substrate temperature of 800 °C is shown in Fig. 4 (series B). It is interesting to note that value of  $4\pi M_s$  first decreases for the oxygen pressure of 0.4 mbar and afterwards increases for higher oxygen pressures. On the other hand, coercivity first increases from 670 to 1290 Oe when oxygen pressure is changed from 0.2 to 0.4 mbar. For higher oxygen pressure, the coercivity decreased to 1100 Oe finally. A similar kind of trend in variation of magnetization and coercivity values has also been reported by Zhou et al. [15] in CFO thin films deposited on single-crystal Si substrates. The observed magnetic properties at different oxygen pressures may be due to decrease in grain size for the films deposited at higher oxygen pressure [21]. The grain size of the film was reduced from 46 to 25 nm for the film deposited at 0.2 and 0.8 mbar (AFM image of the film is shown in Fig. 5). For the lower oxygen pressure, the energy required for the nucleation and grain growth is provided by fewer collisions with the ambient gas upgraded considerably. The oxygen pressure when increased in PLD acts similar to deposition at lower temperature without affecting the surface roughness of the film [22]. The decrease in grain size at higher oxygen pressure therefore leads particles in a single-domain region where coercivity decreases with the decrease in grain size.

Magnetic measurements were also carried out for the films deposited at different laser energy fluences [Series C] and shown in Fig. 6. The film deposited at the lowest energy fluence of 0.65  $\text{J}/\text{cm}^2$  does not show any hysteresis loop indicating that grains do not grow at this energy (no sharp peak is seen in XRD (Fig. 1c) and AFM does not show any grain (Fig. 7a)). Interestingly, the  $4\pi M_s$  value of 2460 G observed for the film deposited at 1.5  $\text{J}/\text{cm}^2$  energy fluence decreases drastically to 625 G when the film is deposited at the highest laser fluence of 3.5  $\text{J}/\text{cm}^2$ . On the other hand, the value of coercivity increases from 670 to 2670 Oe for the laser fluence of 1.5 and 3.5  $\text{J}/\text{cm}^2$ , respectively (The calculated values of  $4\pi M_s$  and  $H_c$  are tabulated in Table 4). It is also seen from the figures that the film deposited at the highest energy fluence of 3.5  $\text{J}/\text{cm}^2$  is hard

**Table 4** The values of magnetization, coercivity, and average grain size of the films deposited at different energy fluences

Energy fluence ( $\text{J}/\text{cm}^2$ )	0.65	1.50	3.50
Magnetization $4\pi M_s$ (Gauss)	No loop	2460	625
Coercivity $H_c$ (Oe)	No loop	670	2670
Average grain size (nm)	–	46	17

**Fig. 7** AFM images of  $\text{CoFe}_2\text{O}_4$  film at an energy fluence of **a**  $0.65 \text{ J/cm}^2$ , **b**  $1.5 \text{ J/cm}^2$ , and **c**  $3.5 \text{ J/cm}^2$



to saturate. The decrease in magnetization may be correlated to the decrease in grain size from 46 to 20 nm when laser energy fluence is increased from 1.5 to  $3.5 \text{ J/cm}^2$  and is revealed by AFM images (Fig. 7). The AFM image of the film deposited for the highest energy fluence of  $3.5 \text{ J/cm}^2$  shows grains of two different contrasts (Fig. 7c), the oblong grains with a smaller size in contrast to the grains with a higher size and an aspect ratio of one. The XRD data of the films deposited at  $3.5 \text{ J/cm}^2$  show only two sharp peaks corresponding to (311) and (440) reflections. The particle size calculated using the Scherrer's formula is also found in analogy to the measured grain size in AFM image. Suzuki et al. [18] have deposited (100)- and (110)-oriented CFO thin films with CCO-buffered STO and  $\text{MgAl}_2\text{O}_4$  substrate by PLD. Similar to our results, the surface structures studied using AFM show oblong grains for the (110). Faster growth along the [110] direction is reasonable due to the fact that the (110) plane face of the copper ferrite unit cell is densely packed. The effect of laser fluence on grain size has been reported in recent part in different oxide thin films deposited by PLD [23, 24]. Shahzad et al. [24] have recently reported that grain size first increases up to a maximum value of  $4 \text{ J/cm}^2$  and decreased for the highest energy fluence of  $5 \text{ J/cm}^2$  in case of superconducting  $\text{BiVPbSr-CaCuO}$  thin films deposited on Si (400) substrates using PLD. It is attributed that at a high laser energy of  $5 \text{ J/cm}^2$ , the atoms aggregated easily with high kinetic energy and

some atoms fragmented after re-sputtering. In our case, the highest energy fluence resulted in poor crystal quality as evidenced by XRD data and thus lowering magnetization.

## 4 Conclusions

In this paper, cobalt ferrite thin films were successfully deposited on fused quartz substrate using pulsed laser deposition technique. The effects of substrate temperature, oxygen partial pressure, and laser energy fluence on structural and magnetic properties have been systematically studied. The films deposited at a lower substrate temperature of  $400 \text{ }^\circ\text{C}$  shows preferential growth of CFO along the [440] direction; however, films deposited at a higher substrate temperature of  $800 \text{ }^\circ\text{C}$  were textured as revealed by XRD data. The coercivity of the film deposited at  $400 \text{ }^\circ\text{C}$  was 1880 Oe, which decreased to 670 Oe for the film deposited at  $800 \text{ }^\circ\text{C}$ . The value of magnetization ( $4\pi M_s$ ), on the other hand, increased from 1940 to 2460 G with the increase in substrate temperature. The value of  $4\pi M_s$  first decreased from for the film deposited at an oxygen pressure of 0.4 mbar and finally increased for the film deposited at higher oxygen pressure. However, coercivity observed to increase from 670 to 1290 Oe when oxygen pressure is increased from 0.2 to 0.4 mbar, which finally decreased to 1100 Oe for an oxygen pressure of 0.8 mbar. It was observed that

the structural and magnetic properties of CFO thin films depend on laser energy fluence. The film deposited at an energy fluence of  $1.5 \text{ J/cm}^2$  shows the maximum value of magnetization which decreases drastically to 625 G for the film deposited at an energy fluence of  $3.5 \text{ J/cm}^2$ . The value of coercivity increases from 670 to 2670 Oe with the increase in laser energy fluence of  $3.5 \text{ J/cm}^2$ . The observed magnetic properties of CFO thin films deposited on fused quartz substrate may be attributed to grain growth and orientational growth of CFO thin films as revealed by AFM and XRD studies and may be useful for possible applications.

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