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Fast laser annealing induced exchange bias in poly-crystalline $BiFeO_3$/Co bilayers

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ABSTRACT

The conventional field cooling process for antiferromagnetic/ferromagnetic bilayer system might strongly damage the interface of $BiFeO_3$ (BFO) with metallic ferromagnetic layer, leading to significant deterioration of exchange bias (EB). In this paper, a field cooling process with fast laser annealing has been proposed and applied on polycrystalline-BFO/Co bilayers, which can effectively modify the EB. In those samples with obvious EB, it is found that the exchange field (H_E) increases abruptly when the laser fluence rises to a critical value, and decreases when the laser fluence is large enough. On the other hand, in those samples with negligible H_E, EB could be easily induced after field cooling with proper laser fluence. In addition, the sign of H_E could also be changed, depending on the direction of the cooling field. In contrast, after field cooling by conventional heat treatment, EB could be neither induced nor enhanced. The feasibility of fast laser annealing accompanied with field cooling to enhance or induce EB in the BFO/Co bilayer can be understood by much less interfacial diffusion in comparison with conventional field cooling.

1. Introduction

Bismuth ferrite ($BiFeO_3$, BFO) is a unique multiferroic material with its ferroelectric and antiferromagnetic phase transition temperatures both well above room temperature [1,2]. However, the antiferromagnetic state of BFO hinders its potential for magnetic storage applications. Fortunately, by virtue of interfacial exchange coupling in a ferromagnet/antiferromagnet (FM/AFM) bilayer, the exchange bias (EB) can be established with a shift (exchange field, H_E) and/or a coercivity (H_C) enhancement of the FM layer's magnetization hysteresis loop. Combined with the ferroelectric property of BFO, the electric manipulation of exchange bias (EB) may be realized in BFO/FM bilayers, which has great potential in new generation spintronic devices with much lower energy consumption [3].

To the best of our knowledge, the EB in ordinary FM/AFM bilayer is often established by two standard methods, i.e. applying a magnetic field during film growth or cooling the sample from above the Néel temperature (T_N) of the AFM material to a low temperature (e.g., room temperature) under a magnetic field after the bilayer is prepared. In order to orient the interfacial magnetic moments and obtain a significant EB effectively, the former is often used in top-pinning structure with AFM layer deposited onto the FM layer, while the latter is used in bottom-pinning structure with AFM layer made beneath the FM layer. In most of the previous EB studies in the BFO/FM bilayers, the EB was achieved on bottom-pinning type bilayers due to that the BFO film has to be deposited at a high temperature for obtaining single phase [4–8]. However, in order to reduce the interface diffusion and/or reaction between the BFO and FM to lower degree, the standard field cooling procedure which is always conducted on bottom-pinning structure has to be abandoned. Instead, a magnetic field needs to be applied during the FM layer deposition. Although this compromised scheme cannot make the reorientation of the interfacial net spins to a much higher degree, fairly strong EB could be still observed in various BFO/FM bilayers, no matter the BFO layer is single crystal [4–6,9–11] or polycrystalline [7,8,12,13].

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Up to date, few studies have been performed by using field cooling (post-annealing) to induce EB or modify EB in the BFO/FM bilayers. Yu et al. [14] found that the EB could be induced in BFO/CoFe bilayer only when the annealing temperature (T_a) was set to lower than 100 °C (i.e. 373 K) and degraded dramatically with further increasing T_a. Besides, Naganuma et al. [9] and our group [13] found that the EB almost disappeared when the measuring temperature was above 400 K, which is much lower than T_N of BFO (~640 K) [1,2]. These results all showed that EB in BFO/FM bilayers cannot live up to a fairly higher temperature albeit much lower than T_N of BFO, which is resulted from severe interfacial diffusion and/or reaction already happened. In order to avoid this problem as far as possible, a rapid heat treatment, e.g. laser annealing, can be considered since it has been successfully used in some traditional FM/AFM bilayer systems, such as NiFe/FeMn [15], NiMn/Co [16], [Pd/Co]/FeMn [17], CoFe/IrMn [18] and so on. In this presented work, we report on the systematic studies of EB on the polycrystalline-BFO/Co bilayers by laser annealing under an applied magnetic field. In comparison with conventional field cooling, this method could effectively enhance or induce the EB in the BFO/FM bilayers.

2. Experiments

Polycrystalline-BFO (80 nm)/Co (4 nm) bilayers were deposited on $LaNiO_3$ buffered silicon substrates by pulsed laser deposition (PLD) combined with magnetron-sputtering technique. Detailed description of the sample preparation can be found in our previous reports [8,13]. The polycrystalline structure and single phase of BFO layer were confirmed by X-ray diffraction (XRD) patterns, which will be addressed later. During the growth of Co layer, if a magnetic field (H_d) of about 200 Oe was applied, obvious EB effect could be observed. Otherwise, no significant H_E could be obtained. A batch of nominally identical samples were fabricated for comparing the effect of field cooling by different means, i.e. laser annealing and conventional heat treatment. As displayed in Table 1, the Co layers of samples A, B, E, F and J were deposited in magnetic field (H_d = 200 Oe), while those of samples C, D, G and H were deposited without magnetic field (H_d = 0 Oe). Note that although the stacking sequences for all the samples were set to be identical, there existed sample to sample difference to some extent due to fluctuation of the fabrication conditions.

Table 1
The fabrication and laser treating parameters for the studied batch of samples from A to J.

Sample	A	B	C	D	E	F	G	H	J
H_d (Oe)	200	200	0	0	200	200	0	0	200
H_{FC} (Oe)	1000	−1000	1000	−1000	1000	−1000	1000	−1000	1000

After the growth of the bilayers, field cooling with laser annealing was used to induce or modify the EB in the samples. The samples were irradiated at different laser fluence ranged from 0 to about 40 mJ/cm^2 by a femtosecond pulsed laser source (Coherent Inc.) with wavelength of 800 nm, repetition rate of 1000 Hz and time duration of 60 fs for each pulse. The sample was irradiated on a spot with diameter of about 600 µm and the irradiation was lasted for only 10 s. After that, the sample was cooled for about 5 min under a magnetic field of about 1 kOe, which is enough to saturate the FM magnetization. The local magnetic-hysteresis (*M-H*) loop within the irradiation area was measured by a longitudinal magnetic-optical Kerr effect (MOKE) magnetometer before and after the laser annealing process. The light source of MOKE magnetometer is a laser diode with wavelength of 405 nm. The MOKE measuring spot with diameter of about only 60 µm is much smaller than the laser irradiation area and nearly at the center, which ensures that the influence of laser annealing can be acquired.

Schematic illustration for the laser irradiation and MOKE measurement can be seen clearly in Fig. 1(a) and (b). Besides the local irradiation on a spot as displayed in Fig. 1(a), Fig. 1(b) shows the spot-by-spot laser irradiation on a whole sample with typical size

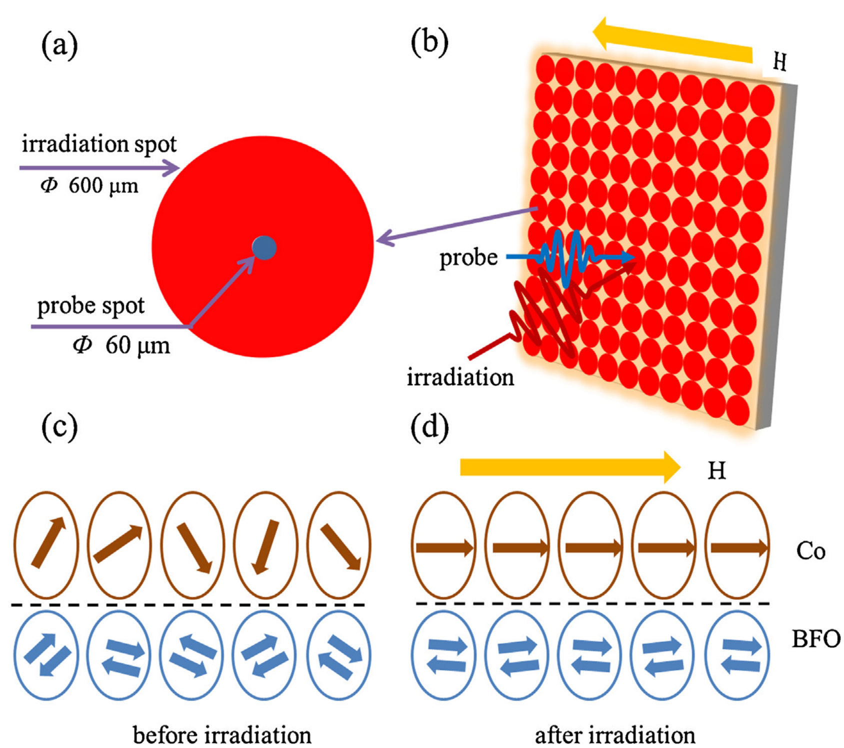

Fig. 1. Schematic illustration for laser irradiation and MOKE detection on a small single spot (a) and a whole sample (b). Schematic illustration for the expected alignment of interfacial FM and AFM domains before (c) and after (d) irradiation under a magnetic field.

of 6 mm × 6 mm. The spot-by-spot irradiation was accomplished by a high precision translation stage, which can control the laser spot moving accurately in an ultimate step of 10 μm. This can ensure every place in the sample irradiated and not twice irradiated as well to the utmost extent. The effect of the laser irradiation under magnetic field is illustrated in Fig. 1(c) and (d). For simplicity, we only consider the case that the Co layer is deposited without magnetic field. Before irradiation, both the FM domains (in Co layer) and the AFM domains (in BFO layer) near the interface are randomly distributed (see Fig. 1(c)). However, after being irradiated under a magnetic field, both FM and AFM domains near the interface are expected to be highly aligned to the cooling field direction, inducing a strong unidirectional anisotropy and establishment of prominent EB, as indicated in Fig. 1(d). In comparison, the conventional field cooling was conducted on a whole sample and the corresponding *M-H* loop was examined by a vibration sample magnetometer (VSM, Microsense EV7), with the annealing temperature varied from 295 K to 525 K. All the *M-H* loops were measured at room temperature with the applied magnetic field (H_a) in the film plane. The cooling field can be either positive (i.e. $H_{FC} = 1000$ Oe) or negative (i.e. $H_{FC} = -1000$ Oe). Positive/negative means the direction of H_{FC} is parallel/antiparallel to the positive directions of H_a and H_d. The values of H_{FC} for all the samples are also listed in Table 1.

3. Results and discussion

Fig. 2 shows the variation of the *M-H* loops with the laser fluence. Four samples, i.e. A, B, C and D, were used through different laser annealing processes and the corresponding results are shown in Fig. 2(a), (b), (c) and (d), respectively. For each figure, the *M*-axis data are the Kerr rotations normalized to those before irradiation. As mentioned above, due to the Co layer deposited with (without) applied magnetic field, EB could (not) be observed in samples A and B (C and D). Deposition in magnetic field can also induce in-plane uniaxial anisotropy in addition to unidirectional anisotropy [19], resulting in the *M-H* loops more rectangular for samples A and B in comparison with samples C and D. Fig. 2 unambiguously shows that the *M-H* loop for the as-deposited sample changes significantly with the laser fluence.

In order to see more clearly how the EB changes with the laser fluence (F_L), the values of H_E and H_C are extracted from Fig. 2 and their dependences on F_L are displayed in Fig. 3. The values of H_E and H_C are calculated by $H_E = (H_{C1} + H_{C2})/2$ and $H_C = |H_{C2} - H_{C1}|/2$, where H_{C1} and H_{C2} denotes the coercive fields at the descending and ascending branches, respectively. The results in Fig. 3(a), (b), (c) and (d) are one-by-one corresponding to those in Fig. 2(a), (b), (c) and (d), respectively. With a positive field applied during laser irradiation, Fig. 3(a) shows that the quantity of H_E keeps almost unchanged when F_L is small and increases abruptly when F_L rises to a critical value of about 2.5 mJ/cm^2. The absolute value of H_E can increase from 21 Oe to more than 32 Oe. In other words, the relative increase of H_E can exceed 50%, indicating that EB can be enhanced significantly with fast laser annealing in the BFO/Co bilayer. With further increasing F_L, the quantity of H_E does not change too much while it begins to decrease when F_L is larger than 20 mJ/cm^2. Different from the behavior of H_E, H_C keeps almost unchanged when $F_L < 20$ mJ/cm^2 and decreases much fast after F_L reaches about 20 mJ/cm^2. The concurrent decreases of H_E and H_C after F_L is raised to be larger than 20 mJ/cm^2 can be understood as weakening of exchange coupling between the Co and BFO layers, which may be caused by interface deterioration under large laser fluence irradiation. With a negative field applied during laser irradiation, Fig. 3(b) displays that H_E decreases in magnitude initially with increasing F_L and changes its sign when F_L is raised to between 20 and 25 mJ/cm^2. However, with increasing F_L, H_C does not change too much firstly and drops abruptly after F_L arrives at about 20 mJ/cm^2, similar to those found in Fig. 3(a). On the other hand, for the as-deposited samples without obvious H_E, Fig. 3(c) and (d) show that EB can be induced effectively when F_L is increased up to about 5 mJ/cm^2, with concurrent increase of $|H_E|$ and H_C. The induced EB exhibits negative (positive) H_E when the applied field is positive (negative) during laser irradiation. However, in these two cases both $|H_E|$ and H_C decrease with increasing F_L when F_L is larger than 30 mJ/cm^2, also suggesting deterioration of interface between Co and BFO layers.

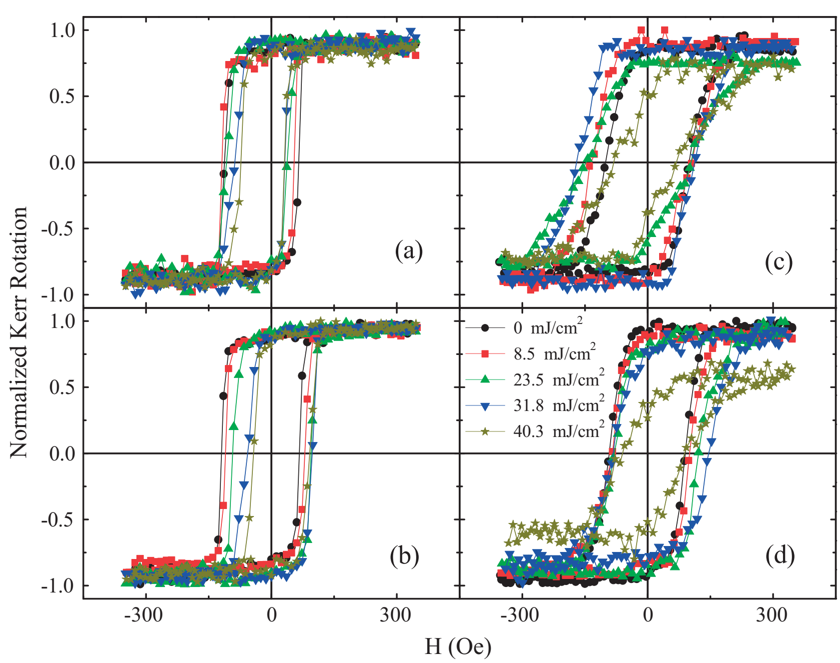

Fig. 2. *M-H* loops measured after different laser fluence irradiation under 1000 Oe for samples A and C ((a) and (c)), and under −1000 Oe for samples B and D ((b) and (d)).

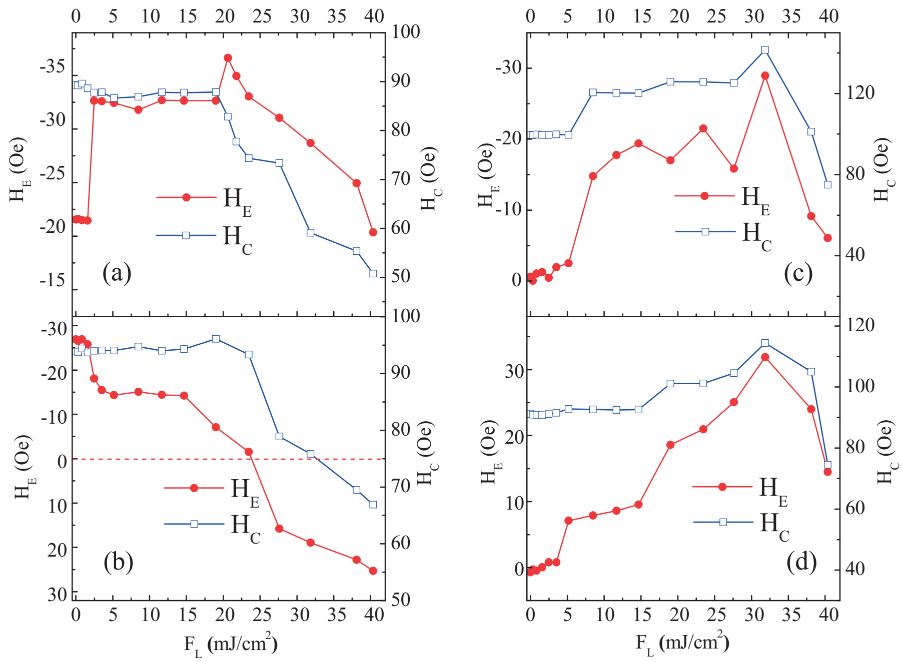

Fig. 3. The laser fluence (F_L) dependences of H_E and H_C for samples A and C irradiated under 1000 Oe ((a) and (c)), and for samples B and D irradiated under −1000 Oe ((b) and (c)). Arrows and dotted lines are guide to eyes.

From the above results shown in Figs. 2 and 3, one can see clearly that EB can be either enhanced or induced by laser annealing under an applied magnetic field. In comparison, the conventional field-cooling was also performed for comparison. After being put into the oven of VSM, the sample was heated to a target temperature (≥300 K) and cooled to room temperature under an applied field in magnitude of 1000 Oe. During the entire process, argon gas was continuously filled into the oven in order to prevent the sample from oxidation. Another series of samples, i.e. E, F, G and H, were used to do the similar operation as those done for sample A, B, C and D. Samples E and F have the similar values of H_E and H_C to those of samples A and B. And samples G and H have negligible EB, similar to samples C and D. Different from the laser annealing, now the heat treatment is conducted on the entire piece of sample. Fig. 4 summaries the dependences of M/M_S, H_E and H_C on the heating temperature (T). On the one hand, Fig. 4 shows clearly that EB cannot be effectively induced on the as-deposited samples with negligible H_E no matter the applied field is positive or negative during field-cooling. On the other hand, Fig. 4 also shows that EB cannot be enhanced and meanwhile the sign of H_E cannot be changed at all on those as-deposited samples with obvious H_E. These results may be caused by partial damage of the sample during the conventional field-cooling, indicated by decrease of M/M_S as well as increase of H_C with increasing the heating temperature. As reported previously [9,13,14], interface diffusion and/or reaction between BFO and FM layer could happen easily if the annealing temperature is higher than about 100 °C (373 K), which will weaken EB dramatically and is in well consistent with our findings.

Therefore, in comparison with conventional field-cooling, EB can be effectively enhanced or induced by fast laser annealing in the BFO/Co bilayer samples. This indicates that the BFO-Co interface has not been seriously damaged during a short period (10 s) of laser irradiation with proper fluence, whereas the interfacial coupling between BFO and Co layers has been strengthened or established in the meanwhile. To the best of our knowledge, laser irradiation was also performed on conventional AFM/FM bilayers to enhance or modify the EB [15–17]. For example, Mohanan et al. [16] found that H_E could be increased by laser irradiation with proper laser fluence on NiMn/Co layers. They attributed these results to increased grain size and improved (111) texture of the NiMn layer after laser irradiation, which was verified by XRD characterizations. Whether or not the same mechanism can be applied to the present polycrystalline BFO/Co bilayers, further structural examination is necessary and will be addressed in the following.

For the BFO/FM bilayers with single-crystal BFO film employed as pinning layer, it has been found that EB is very strong with (001) [4,5,9,11] or (110)-orientated [6] BFO layer and becomes much weaker if the BFO layer is (111)-orientated [6]. However, for the

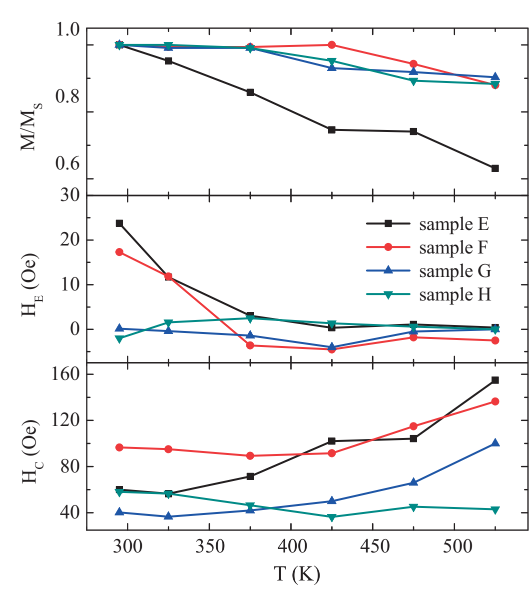

Fig. 4. Annealing temperature (T) dependences of (a) M_S/M, (b) H_C and (c) H_E for samples E and G cooled under 1000 Oe and for samples F and H cooled under −1000 Oe by conventional heat treatments.

polycrystalline-BFO/FM bilayer system, because EB has not been widely studied yet, the influence of BFO texture on EB still remains unclear. Our previous studies show that the polycrystalline BFO film has the strongest XRD peak around 32°, which is indexed as 'B (110)' [8]. The subsequent results (unpublished) show that EB is strongly correlated to the intensity of the main peak around 32°. Generally, the stronger the intensity of the 32°-peak is, the more significant EB can be observed in the BFO/FM bilayers. Although this scenario is much helpful for us to obtain obvious EB in polycrystalline-BFO/FM bilayers, it is very hard to well control the BFO layer texture technically because it is determined by too many growth parameters, e.g. the substrate temperature (T_S) [20]. The temperature fluctuation between different runs of BFO deposition may be a major factor to determine the intensity of the 32°-peak and thus results in the EB difference from sample to sample.

In order to study the variation of BFO layer texture after laser irradiation and its influence on the EB in the BFO/Co layer, sample J was chosen to do the work. Although the stacking sequence for sample J is identical to all those samples mentioned previously, the texture for BFO layer in sample J is prominent with much stronger 32°-peak in the XRD pattern as shown by the black line in Fig. 5(a). Therefore, the EB is very significant with H_E = 55 Oe for sample J and the corresponding *M-H* loop measured by VSM can be seen by the black line in Fig. 5(b). Because sample J has to be irradiated completely for further magnetic and structural characterizations and the laser irradiation spot is very small (~600 μm) as well, it was therefore irradiated spot-by-spot as sketched in the right panel of Fig. 1 and for each spot the irradiation repeats the operation as that in the left panel of Fig. 1. After the whole sample irradiation (F_L = 5.1 mJ/cm^2) under 1000 Oe was finished, the *M-H* loop was re-measured by VSM, as exhibited by the red line in Fig. 5(b). It shows clearly that H_E increases from 55 Oe to 70 Oe, i.e. the relative increase amount is nearly 30%. Similar to the MOKE results in Figs. 2(a) and 3(a), H_C keeps almost unchanged after irradiation,

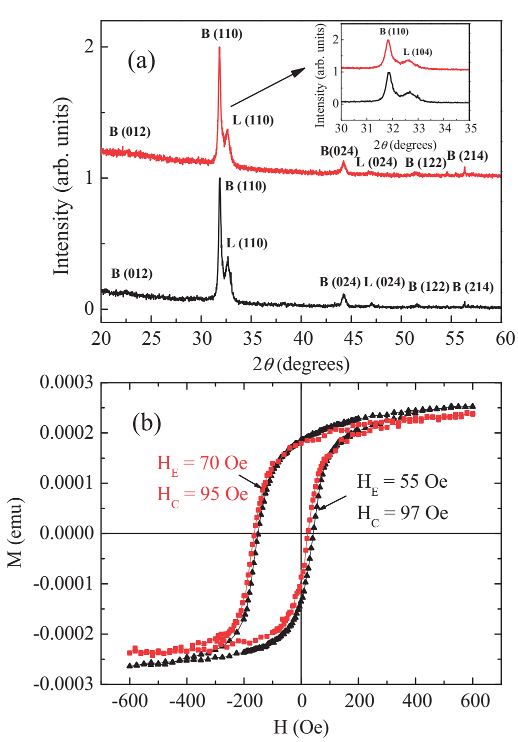

Fig. 5. XRD patterns (a) and *M-H* loops (b) obtained for sample J before and after laser annealing on the whole sample under 1000 Oe. The enlarged XRD patterns around 2θ = 32° are shown in the inset of (a). 'B' and 'L' denote $BiFeO_3$ and $LaNiO_3$, respectively.

indicating that the *M-H* loop shifted leftward entirely. In addition, a slight decrease of M_S after irradiation suggests that weak damage to the BFO-Co interface may still happen. These above results unambiguously show that the EB for sample J has been enhanced significantly after it was irradiated thoroughly under a positive magnetic field. However, as exhibited by the red line in Fig. 5(a), the XRD pattern has almost remained unchanged, even in the vicinity of the 32°-peak shown in the inset of Fig. 5(a). In addition, it also needs to be noted that no obvious impurity diffraction peaks can be observed in Fig. 5 before and after the sample was irradiated.

Therefore, different from those in conventional FM/AFM bilayers, the laser-irradiation caused EB enhancement is not due to improved texture of BFO in the BFO/Co bilayer. As mentioned in the 'Experiments' part, the BFO layer needs to be deposited at a high temperature (generally about 700 °C) for achievement of high quality single phase. Therefore, it is reasonable that the fast laser annealing done in ten seconds is unlikely to make the sample rise quickly to a high temperature enough to enlarge the grain size and modify the texture of BFO film consequently. On the other hand, if the temperature is increased up to a much high value (e.g. 700 °C) after the sample is irradiated, the interdiffusion and/or reaction between BFO and Co layers would happen seriously and thus result in dramatic degradation of EB, as those observed in the conventional field-cooling experiments [9,13,14]. However, these anticipated results have not been found in the laser-annealed BFO/Co bilayers. Because the instant temperature within the irradiated area is quite difficult to be measured spot-by-spot, it becomes very hard to reveal the hidden mechanism responsible for EB enhancement by field cooling with laser annealing. According to our point of view, the laser annealing and conventional field cooling may affect the exchange bias in the same mechanism. That is to elevate the sample temperature above the Néel temperature and below a much higher one at which the texture could be modified, and obtain a well-aligned interfacial exchange coupling in magnetic field. However, the large cooling ratio in laser annealing due to 10-s of time irradiation can avoid the interfacial diffusion significantly, which is inevitable for conventional field cooling. Finally, it needs to be noted that the above enhanced or induced EB generated by laser annealing can only be observed with proper laser irradiation fluence. If the laser fluence is too low, interfacial BFO spins would be inert and take no response to the external field, leading to unchanged EB. On the contrary, if the laser fluence is too high, the atoms near the BFO-Co interface would become very active, resulting in serious mutual diffusion and deteriorating the interface. Therefore, EB will be weakened dramatically, which is in good agreement with our experimental findings.

4. Conclusions

In summary, fast laser annealing under external magnetic field has been investigated extensively in the polycrystalline-BFO/Co bilayer samples, which were made by PLD combined with magnetron sputtering. Exchange bias effect could be enhanced (or induced) in those samples with (or without) EB when the laser fluence was set to an appropriate value. In comparison, field cooling with conventional heat treatment was not applicable. By excluding the origin of the BFO texture modification after irradiation, these results are mainly due to that the fast laser annealing can effectively inhibit the interface diffusion between the BFO and Co layers during field cooling in comparison with conventional heat treatment. The experimental findings of the present work provide an effective method to induce or enhance the exchange bias effect locally in micrometer size (even down to nanometer size) in BFO-based multilayered structure, which might be important for the applications of multiferroicity in spintronic devices.

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