Cooperation effect of indium and vanadium co-doped into bismuth-iron garnets on magnetic properties

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Abstract
For achieving tunable saturation magnetization and line width, different cations were doped into the YIG-based garnets, which the standard molecular formula could be expressed as A₃[Fe₂₋ₓMₓ]Fe₃₋₀.₁₄(Nₓ₋₀.₁₄)O₁₂. Most researchers have reported a single rule of one element doping (x or y) on its magnetic properties. However, the cooperation effect of x + y to the magnetic contribution was not clearly clarified. In this work, multi-doped bismuth-iron garnets {Biₓ₀.₈₅Ca₂.₁₆}[Feₓ₋₁.₇₂₋₀.₁₄]Fe₃₋₀.₁₄(Nₓ₋₀.₁₄)O₁₂ (x = 0.72–0.14 and y = 1.24–1.38) with low saturation magnetizations (4πMₛ = 200–600 Gs at 298 K) were prepared by a conventional fluxing agent method. The cooperation effect of In³⁺ and V⁵⁺ co-doped into bismuth-iron garnets (BIG) on their structures and magnetic properties were systematically investigated using XPS, TEM and VSM. It could be found that the total doped concentration (x + y) was decreasing regardless of the increasing V concentration (y) and decreasing In concentration (x). As-synthesized BIG presented a well single-crystal structure, and the lattice spacing was decreasing with the decrease of x + y in accompanying with the transition of dislocations from point defects to edge dislocations. Both linear increase of Curie temperature and 4πMₛ with the decrease of x + y was revealed. The mechanism could be attributed that the doped non-magnetic ions could reduce the average nearest-neighbor coordination ions for oxygen ions and weaken the anti-ferromagnetic super-exchange interactions among the magnetic ions within the structure, namely dilution effect. By comparison, the total concentraions were higher than that of previous works, which the tailorable 4πMₛ of ferrite was not reported. Here we revealed the controllable 4πMₛ with higher total concentrations (x + y ≥ 1.52). These findings will be provided more opportunities for applications in microwave devices.

1. Introduction
Yttrium iron garnet (Y₃Fe₅O₁₂, YIG), a classical ferrimagnetic material, possesses numerous amusing electromagnetic properties at microwave region, such as the tailorable 4πMₛ, tunable Curie temperature (Tₛ) and narrow ferromagnetic resonance linewidth [1–6]. These fantastic electromagnetic properties have caught massive attentions and the YIG-based crystal materials were widely applied in microwave devices, involving filter rejector, oscillator, limiter and so on [7–12]. In the YIG crystal cell, the Y³⁺ ions occupy the special position 24c with dodecahedral coordination, the Fe³⁺ ions occupy the 24d (termed Fe-24) and 16a (termed Fe-16) sites with three tetrahedral and two octahedral sites; and all of the oxygen ions are in the general 96h position [13]. Generally, the electromagnetic properties of YIG system mainly originate from the Fe³⁺ ions. To adjust the electromagnetic properties of YIG-based materials, different cations (V⁵⁺, Mn²⁺, Al³⁺, In³⁺, etc) and their combinations are doped into the YIG system to replace the different sites of Fe³⁺ (Fe-24 and Fe-16) [11–16]. Kim...
et al. have reported the decreased saturation magnetization of Al doped Fe-24 in YIG with the increase of doped concentrations [12]. Besides, many investigations reported that doping In\textsuperscript{3+} and V\textsuperscript{5+}, substituted the Fe-16 and Fe-24 positions separately, could both tune the value of 4πM\textsubscript{s}. [11, 16].

Aiming at explaining the mechanism of tunable 4πM\textsubscript{s} for the doped YIG materials, most scholars carried out a formula: \( M_s = |m_{c} - (m_{d} - m_{a})| \), where the \( m_{c} \), \( m_{d} \) and \( m_{a} \) represents the magnetic moment of dodecahedral Y ions, tetrahedral 24d and octahedral 16a Fe ions, respectively [11–13]. Based on this equation, the values of \( m_{d} \) and \( m_{a} \) are determinedly responsible for modifying the 4πM\textsubscript{s}. Thus lots of studies focused on the changes of magnetic moment at Fe 24d and 16a positions according to dope the transition metal ions. Such as the \{Y\textsubscript{1−x}Gd\textsubscript{x}O\textsubscript{12},(Fe\textsubscript{1−y}In\textsubscript{y})\textsubscript{O\textsubscript{12}}\} samples, the 4πM\textsubscript{s} was attributed to the changed \( m_{a} \) of Fe-16a with different amount of In\textsuperscript{3+} as fixing a doped V\textsuperscript{5+} concentration at Fe-24d [11]. Besides, with a fixed In\textsuperscript{3+} substitution in Fe-16a site, the magnetic properties of 2Ca\textsuperscript{2+}−V\textsuperscript{5+}−In\textsuperscript{3+}−doped-YIG ferrites also could be remarkably influenced by V\textsuperscript{5+} doping, and performed a decreased 4πM\textsubscript{s} with the increased amount of V\textsuperscript{5+} from 0 to 0.8 [16]. These previous works have demonstrated that a single element substituted one of Fe position as fixing the other position (24d or 16a) could induce the change of 4πM\textsubscript{s}. In the standard molecular formula of doped YIG-based materials, \( A_{3}\text{[Fe}\textsubscript{2−x}M_{x}\text{]}\text{(Fe}_{x}\text{−y}\text{M}_{y})\text{O}_{12} \), where the \( A \) represents the Bi, Gd, Ce, Ca, etc; \( M \) and \( N \) represent the different transition metal ions, such as V\textsuperscript{5+}, Mn\textsuperscript{3+}, Al\textsuperscript{3+}, In\textsuperscript{3+}, etc, the researchers only reported a single rule of the \( x \) or \( y \) effect on the 4πM\textsubscript{s} [11–15]. Unfortunately, the cooperation effect of \( x + y \) to the contribution of 4πM\textsubscript{s} was not clearly clarified.

Fluxing agent method is a low-cost, simple, and environment-friendly preparation technique. Its greatest advantage is that the crystallization process can be carried out at atmospheric pressure, and crystals can be produced at a temperature lower than the corresponding melting point. The flux method can make the crystal subjected to less mechanical stress and can obtain high-quality crystals. In previous studies [11, 16], the YIG-based single-crystals with large sizes and high-qualities were always fabricated by the flux method. In this work, the In\textsuperscript{3+} and V\textsuperscript{5+} co-doped YIG samples with high concentrations were prepared by the fluxing agent method. After the magnetic properties measure, the tuned 4πM\textsubscript{s} with the decease of the total doped concentration (\( x + y \)) was performed. By co-doping In\textsuperscript{3+} and V\textsuperscript{5+} ions into the position of Fe 24d and 16a, we revealed the effect of cooperation of \( x + y \) on the 4πM\textsubscript{s}.

2. Methods

Molten PbO and Bi\textsubscript{2}O\textsubscript{3} fluxing agents were used in the growth procedure. The other materials (CaCO\textsubscript{3}, Fe\textsubscript{2}O\textsubscript{3}, V\textsubscript{2}O\textsubscript{5} and In\textsubscript{2}O\textsubscript{3}) for a batch weight of approximately 1000 g were mixed in a mortar and pestle, screened, and packed into a 1200-cc platinum crucible. A platinum lid was covered over the crucible, retarding volatilization of the flux. In the processes of high temperature melting, the weight loss was mainly ascribed that the CaCO\textsubscript{3} were broken down and released CO\textsubscript{2}, while the weights of other materials (PbO, Bi\textsubscript{2}O\textsubscript{3}, Fe\textsubscript{2}O\textsubscript{3}, V\textsubscript{2}O\textsubscript{5} and In\textsubscript{2}O\textsubscript{3}) were almost unchanged. The purities of all raw materials were greater than 99.99%. Best results have been obtained when an initial 4-h soak at 1200 °C is used. Temperatures higher than 1200 °C tended to reduce the quality of the crystals. The furnace was cooled at 2 °C h\textsuperscript{−1} to an intermediate temperature of approximately 1120 °C and held for 16 h. This hold-period is believed to promote equilibrium conditions in the melt prior to the crystal growth. The crystals were grown from melts, omitting the holding period. The prepared crystals contained large flux inclusions, indicating rapid growth from a supersaturated solution. After the hold-period, the crucible is again cooled at 2 °C h\textsuperscript{−1} to 1050 °C. During the processes, it was removed from the furnace and the flux poured off. The crystals are cleaned from any remaining flux with boiling 1:1 HNO\textsubscript{3}:H\textsubscript{2}O. According to tune the molar ratios of V\textsubscript{2}O\textsubscript{5} and In\textsubscript{2}O\textsubscript{3}, the different doped concentrations (\( x \) and \( y \)) were obtained, which are marked as 1#–
In addition, by means of the x-ray photoelectron spectroscopy (XPS), x-ray diffraction (XRD), transmission electron microscope (TEM) and vibrating sample magnetometer (VSM), the relevant composition, crystalline quality and magnetization of varied samples were observed.

### 3. Results and discussion

As the XPS spectra of 1# BIG sample (figure 1), the peaks of Bi, Ca, In, V, O and Fe could be attained. To clarify the corresponding valence of each element in the samples, the relative peaks of each element were fitted, as

| Term | Bi$^{3+}$ (%) | Ca$^{2+}$ (%) | Fe$^{2+}$ (%) | Fe$^{3+}$ (%) | In$^{3+}$ (%) | V$^{5+}$ (%) | O$^{2-}$ (%) |
|------|---------------|---------------|---------------|---------------|---------------|---------------|---------------|
| 1#   | 6.63          | 17.31         | 5.49          | 20.96         | 6.3           | 10.8          | 32.21         |
| 2#   | 6.67          | 17.32         | 6.05          | 21.49         | 4.56          | 11.35         | 32.24         |
| 3#   | 6.68          | 17.39         | 6.15          | 21.91         | 3.97          | 11.63         | 32.27         |
| 4#   | 6.7           | 17.42         | 6.35          | 22.77         | 2.59          | 11.94         | 32.23         |
| 5#   | 6.72          | 17.45         | 6.51          | 23.42         | 1.93          | 12.13         | 32.12         |
shown in figure 2. The fitted peak located at 158.78 and 164.08 eV corresponds to the binding energy of Bi 4f5/2 and Bi 4f7/2 (figure 2(a)), respectively, indicating the existence of Bi3+. For the Ca atoms (figure 2(b)), the fitted results are situated at 346.38 and 349.88 eV, which reveals the 2+ valence of Ca. In figure 2(c), the In 3d peaks at 444.08 and 451.68 eV ascribes the In 3d5/2 and In 3d3/2, respectively, suggesting that the valence of In is 3+. For the curves of V in figure 2(d), the V 2p spectra exhibit doublets with the banding energies at 516.88 and 524.38 eV, belonging to the V 2p1/2 and V 2p3/2, respectively, and revealing the existence of V5+. The fitted O peaks at 529.78 and 531.48 eV correspond to the orbits of O 2p, showing the 2- valence of O element (figure 2(e)). In the Fe case, the peaks at 710.28, 712.58, 718.28 and 723.78 eV are related to the octahedral Fe2+ 2p3/2, Fe3+ 2p3/2, Fe2+ 2p1/2 and tetrahedral Fe3+ 2p1/2, respectively. The existence of Fe5+ in the YIG system is unavoidable, which have been demonstrated by the reported works [17]. Besides, the related XPS results of other samples (2#–5#) are shown in the supplement materials (figures S1–S4 (available online at stacks.iop.org/MRX/8/016104/mmedia)). The relevant component ratio of each elements is analyzed in table 1. After a normalization processing, the corresponding values of x, y and x + y are shown in table 2. Obviously, the total doped ions concentration (x + y) is decreasing regardless of the increasing V concentration (y) and decreasing In concentration (x). Here the total concentrations of x + y are all higher than that of other works [11, 13, 16]. At this point, the influence of high dopants to the magnetization properties of doped YIG-based samples are still controversial.

HR-TEM images of all samples are shown in figure 3. The well-defined lattice fringes are shown, demonstrating the great crystallinity of the samples. Besides, the measured inner planar spacing of ~0.279 nm to all samples is consistent with the distance of the (420) plane to BIG. Noticeably, the measured inner planar spacing is gradually decreased from 0.279 to 0.276 nm with the decrease of x + y. The corresponding ion radius of the In3+, V5+ and Fe5+ ion is 0.093, 0.054 and 0.065 nm, respectively [11, 18]. With the decrease of In3+ ions and increase of V5+ ions, the crystal cell is contracted, leading to the decreased inner planar spacing from 1# to 5#. Combined with the relative fast Fourier transform (FFT) results of each samples, as shown in the insets of figure 3, the clear diffraction spots were observed, indicating that the cubic garnet is a great single crystal. In figure 4, the HR-TEM dislocation images of varied samples are displayed. Obviously, different dislocations exist in the doped products. For the higher x + y concentrations (1# and 2#), there are mainly some point defects, which results from the substitution of ions by In3+ and V5+ ions. With the decrease of

| Sample | x   | y   | x + y |
|--------|-----|-----|-------|
| 1#     | 0.72| 1.24| 1.96  |
| 2#     | 0.52| 1.32| 1.84  |
| 3#     | 0.44| 1.33| 1.77  |
| 4#     | 0.30| 1.37| 1.67  |
| 5#     | 0.14| 1.38| 1.52  |

The values of Inx, Vy and x + y.

Figure 4. HR-TEM dislocation image of (a) 1#, (b) 2#, (c) 3# and (d) 4#, respectively.
dopants, the type of dislocations transits to edge dislocations, which could be assigned to the lattice contraction as the decrease of $x + y$. Redwing et al. reported that the density of edge-type dislocations increased with increasing $x$ in Si-doped Al$_{x}$Ga$_{1-x}$N epilayers [19]. Additionally, Yuan et al. found that the different Nb-doping concentration could induce the transformation from point defects to edge dislocations in TiAl alloys [20]. Thus changing the concentrations of doped ions could make the various dislocation types.

Figure 5 (a) shows the $M$-$T$ curves of samples. A sharp drop at the region from 400 to 500 K could be attained, meaning the ferrimagnetic-paramagnetic transition temperature, namely $T_c$. After a derivation analysis of $M$-$T$ curves, as shown in the inset, the $T_c$ for the $1$–$5$ is 424.8, 435.7, 449.7, 479.1 and 488 K, respectively. It performs a linear increase tendency with the decrease of $x + y$, which could be attributed to the substitution of non-magnetic In$^{3+}$ and V$^{5+}$ ions. In this BIG structure, the ferrimagnetic coupling of ferric ions is via an electron trapped in the bridged oxygen ions, also termed as super-exchange interaction [11–13, 21]. The substitution of non-magnetic ions could dilute the ferrimagnetic coupling of ferric ions, leading to the decreased spin moment of samples further. Hence, the increased Curie temperature of samples with the decreased doped concentration ($x + y$) is disclosed by the dilute ferrimagnetic coupling of ferric ions [11, 21].

The $T_c$ of the samples increased almost linearly from 424.8 K ($x + y = 1.96$) to 488 K ($x + y = 1.52$). Compared with the pure YIG ($T_c = 560$ K), the lower Curie temperature could ascribe the substitution of non-magnetic In$^{3+}$ and V$^{5+}$ ions [22]. Such a linear increase of $T_c$ may be attributed to the weakened dilution of magnetic ions in the BIG structure. This ‘dilution effect’ is supposed to reduce the average next nearest-neighbor coordination number $z$ for any magnetic ions through oxygen ions and thus weaken the anti-ferromagnetic super-exchange interactions among the magnetic ions within the structure, as indicated by the formula [11]:

$$T_c = \frac{2zs(s + 1)}{3k_B},$$

where $J$ is the exchange integral, $s$ the spin quantum for magnetic moment, $k_B$ the Boltzmann constant and $z$ the average coordination number of magnetic ions through oxygen ions for each magnetic ion as a center.
In the YIG system, the dominated magnetic coupling is the super-exchange interactions of Fe-24, Fe-16 and O ions. Typically, as shown in the figure 5(b), in the super-exchange mode, an electron of O^{2-} 2p orbit would jump to the 3d orbits of a nearby Fe^{3+}, and the other one would couple with an electron of nearby Fe^{3+} ion.

Based on this, an anti-ferromagnetic super-exchange interaction is formed. When the Fe^{3+} ions are substituted by the doped In^{3+} and V^{5+} ions at a low amount, the In^{3+} and V^{5+} ions would replace the Fe-16 and Fe-24 regularly. Therefore, the doped non-magnetic ions could reduce the average nearest-neighbor coordination ions for oxygen ions and weaken the anti-ferromagnetic super-exchange interactions among the magnetic ions within the structure (also termed as dilution effect), leading to the increased 4π\(M_s\).

The M-H curves of all samples at room temperature measured by VSM are shown in figure 6(a). The 4π\(M_s\) is 182 (1#), 293 (2#), 393 (3#), 495 (4#) and 592 Gs (5#). It is clear that the 4π\(M_s\) is increasing with the decrease of x + y. Actually, the magnetism of YIG-based ferrite mainly comes from the super-exchange coupling between the Fe^{3+} ion in tetrahedral d position and Fe^{3+} ion in octahedral a position. Because of this super-exchange coupling effect in YIG, the magnetic moment of Fe^{3+} ion in octahedral a and the magnetic moment of Fe^{3+} ion in tetrahedral d present parallel and opposite directions, as shown in figure 5(b). Since octahedral and tetrahedral sublatices antiferromagnetically coupled, when the dilution occurred in both octahedral and tetrahedral sublatices, it could cause the increase of magnetization. Here the 4π\(M_s\) is increasing with the decrease of x + y concentration, indicating the dominant role of dilution effect. With the high concentrations (>1.3), the misplacement or migration of In^{3+} and V^{5+} ions between tetrahedral and octahedral sites might be accured. For revealing the realistic lattice occupation of doped ions in the structure, especially high doping amount, most researches selected the x-ray diffraction intensity ratios I(422)/I(420) of the polycrystalline YIG-based ceramics [11, 23]. In this work, the In^{3+}/V^{5+} co-doped garnets are single crystals, and the XRD pattern shows only one diffraction peak of (400) (see supplement materials, figure S5). So it is hard to determine the accurate lattice occupation of In^{3+} and V^{5+} ions according to the XRD intensity ratios. Aiming at the location of In^{3+} and V^{5+} in lattice cells, it should be adopted more advanced detection equipment, such as synchrontron radiation, which will be carried out in the future. To disclose the mechanism of the prepared doped productions, the cooperation of magnetic coupling and the number of magnetic ions in the doped YIG system must be employed.

In order to demonstrate the analysis mentioned above, the 4π\(M_s\) in this work and past reported works are displayed in figure 6(b). For the work of Guo et al in a same substitution of Fe-24 site in In^{3+}-doped-YIG samples, as the concentration of In^{3+} and total doped increased from 0 to 0.9 and 0.85 to 1.75, respectively, they attained the nonlinearly changed 4π\(M_s\) of doped YIG ferrites [11]. When the total concentration is lower than 1.55, their 4π\(M_s\) was increased, whereas exceeding the critical concentration 1.55, the 4π\(M_s\) was decreased. As the V^{5+} increased from 0.3 to 0.8 and total doped concentration changed from 1.0 to 1.5 of YIG-doped ferrites, Winkler et al also reported the nonlinear 4π\(M_s\) result, which showed an increased tendency under 1.3 and decreased tendency over 1.3 [16]. In addition, Dionne et al also showed the decreased magnetization of 2Ca^{2+}•V^{5+}•In^{3+}−doped-YIG when the total doped concentration is higher than 1.3 [22]. Based on this, we could find that all the reported investigations of tunable 4π\(M_s\) with varied total doped concentration displayed an inflexion point at 1.3 − 1.55. Nevertheless, there are almost no systematic studies about the YIG-based samples with the doped amount higher than the inflexion point concentration. Combined with the total concentration in this work, the lowest doped concentration is x + y = 1.52, locating at the critical concentration range (1.3−1.55), which the value of 4π\(M_s\) = 592 Gs is much closer to other works [11, 21]. As the increase of doped concentration further, the magnetic property of BIG samples with high doped concentration was investigated deeply. Consequently, the increased 4π\(M_s\) of the doped samples in this work could be assigned to the decreased magnetic ions. By comparison, the total concentrations were higher than that of previous works [11, 16, 21], which the tailorable 4π\(M_s\) of ferrite was not reported. Here we revealed the controllable 4π\(M_s\) with higher total concentrations (x + y ≥ 1.52). These findings could be provided more opportunities for applications in microwave devices.

### 4. Conclusions

Using the fluxing agent method, we fabricated doped BIG single-crystals, which the standard molecular formula could be expressed as [Bi_{0.84}Ca_{2.16}]\{Fe_{2-\text{In}}\}_{\text{Fe}3\text{−V}_{\text{y}}}\text{O}_{12}. According to XPS testing, the total doped ions concentration (x + y) was decreasing with the increase of V concentration (y = 1.24−1.38) and decrease of In concentration (x = 0.72−0.14). From TEM analysis, the lattice cell was shrunked with the decrease of x + y. Based on the M-T and M-H data, the Curie temperature and 4π\(M_s\) were both increasing. The magnetic mechanism originated from the non-magnetic ions of In^{3+} and V^{5+} was revealed by the weaken super-exchange interactions. In the present studies, we elaborated systematically the cooperation effect of x + y to the magnetic contribution, and made up the tunable 4π\(M_s\) at higher total concentrations (x + y ≥ 1.52). These results can guide the development of BIG-based materials for applications in microwave devices.
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Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

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