

Article

Preparation of Chain-like CoBiNi Alloy as Soft Magnetic Materials for High Permeability and Low Loss

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Abstract

5G communication commercialization is accelerating in many countries. At present, a large number of communication materials are deployed to transmit millimeter waves for 5G base stations. However, it brings huge energy consumption due to the shortcomings of the current materials. Therefore, a novel soft magnetic material with high magnetic permeability and low dielectric constant is urgently needed to reduce the energy loss of 5G base stations. In this work, a series of CoBiNi alloys were prepared using the hydrothermal reduction method, with bismuth (Bi) as the dopant. The results indicate that Bi can regulate the magnetic permeability of soft magnetic materials; the permeability of the Co₂₀Bi₅Ni₇₅ alloy fluctuates stably around 1.50 within the frequency range of 14.00–18.00 GHz. The saturation magnetization exhibits an upward trend with increasing Bi doping, with the Co₂₀Bi₅Ni₇₅ sample reaching a saturation magnetization of 73.11 emu/g. The coercivity and residual magnetization characteristics confirm that Co₂₀Bi₅Ni₇₅ is a typical soft magnetic material. The microwave return loss (RL) of the Co₂₀Bi₅Ni₇₅ alloy was consistently higher than −6.89 dB across the 1.00–18.00 GHz frequency range when the sample thickness was 5 mm. The increased magnetic permeability of the Co₂₀Bi₅Ni₇₅ alloy is attributed to the ability of Bi³⁺ to suppress carrier migration, thereby increasing the resistivity of the crystal structure and consequently improving the material's magnetic permeability. These findings provide new insights into the preparation of high-permeability soft magnetic materials.

Keywords: chain-like structure; CoBiNi alloy; high magnetic permeability; low magnetic loss; magnetic dielectric properties



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1. Introduction

In today's Internet of Things era, the development of 5G communication drives the explosion of mobile data traffic, cloud services and hyper-realistic media enabled by big-data analysis [1–3]. Compared with 4G communication, the 5G millimeter-wave communication operates at a higher frequency band and has weaker diffraction capability, resulting in significantly increased base station energy consumption. As a result, conventional electromagnetic materials can no longer balance signal transmission efficiency with low energy consumption requirements [4–7]. Therefore, developing novel soft magnetic materials with high permeability and low magnetic loss is the core key to achieving efficient and

low-power operation of 5G base stations. In order to realize low-power signal transmission in 5G-based stations, the soft magnetic materials simultaneously need to meet four basic properties of low dielectric constant, low dielectric loss, high magnetic permeability and low magnetic loss as far as possible. Among these, permeability and magnetic loss are the two core indicators that determine the energy efficiency of 5G communication transmission.

The magnetic permeability of soft magnetic materials may be achieved by optimizing their intrinsic material parameters, thereby overcoming Snoek's limit. In recent years, a significant amount of research has explored various feasible methods for achieving this breakthrough through the modulation of relevant parameters [8–12]. Ceramic ferrites as magnetic cores have been widely used in high-power and high-frequency inductors due to their low cost and relatively low magnetic losses around 100 KHz [13,14]. However, conventional ferrite soft magnetic materials are constrained by the Snoek limit, resulting in low permeability at high frequencies, making them unsuitable for high-frequency application scenarios in 5G.

In contrast, CoNi-based metallic alloys offer advantages such as high saturation magnetization, tunable permeability, and easily controllable microstructure, making them highly promising candidate materials for high-frequency soft magnetic applications [15,16]. Kurlyandskaya et al. found that the CoNi alloy has a wide range of physical and chemical properties in the fields of microwave absorption and magnetic resonance imaging agents. The applications of CoNi alloys depend on two factors: composition and morphology, which together determine their intrinsic magnetic moment, magnetic permeability and magnetic properties [17–21]. Liu et al. [9] synthesized CoNi alloys of different shapes by using surfactants as structure-directing, the results indicated that surfactants play a crucial role in enhancing the magnetic permeability of cobalt–nickel alloys.

Moreover, saturation magnetization (M_s) is an internal parameter of the material, which is determined by the number of unpaired electrons outside the core of the soft magnetic materials. Meanwhile, the M_s values of multiphase materials are usually higher than those of monophase materials [22,23]. For example, Fe-Co, Fe-Ni, and Fe-Si-Al composites have higher M_s values than the monophase materials such as Fe, Co, Ni, Si, and Al. Importantly, the doping of critical metals may significantly enhance the magnetic permeability of alloy materials. Current research indicates that dopants such as Al, Cr, V, Bi and rare-earth ions are employed in CoNi-based ferrite systems. In particular, Bi^{3+} has a noticeably larger ionic radius (1.17 Å at a coordination number of 6), which is far greater than that of Fe^{3+} and other transition metal ions [24]. Where Bi^{3+} primarily substitutes for Fe^{3+} at the octahedral B sites in the spinel lattice, this size mismatch introduces local lattice distortion and alters the cation distribution [25]. Furthermore, contrary to the expectation that the dilution of non-magnetic Bi^{3+} would simply reduce the magnetic susceptibility, an appropriate amount of Bi^{3+} substitution can improve magnetic permeability through microstructural optimization. Sertkol et al. (2022) found that Bi^{3+} -substituted $\text{Co}_{0.5}\text{Ni}_{0.5}\text{Bi}_x\text{Fe}_{2-x}\text{O}_4$ nanofibres exhibited enhanced coercivity and rectangularity at $x = 0.04$, indicating an improved domain structure [24]. Similarly, Alqarni et al. (2022) reported that in a Co-Ni spinel ferrite synthesized via hydrothermal synthesis, Bi^{3+} doping significantly modified the magnetic domain configuration; the coercivity of the samples was tunable within the range of 101 to 1038 Oe depending on the Bi content, and $\text{Bi}_{0.04}$ doping led to the formation of a single-domain structure with uniaxial symmetry, a configuration that enhances magnetic permeability [26].

In addition, Mohammad et al. prepared thin films of luteium-iron garnet substituted by bismuth element (Bi) with low coercivity. It has a wide range of potential applications in next-generation integrated optics, magneto-photonics and magnetic field sensors [27,28]. In addition, Bi atoms could inhibit the movement of charge carriers and increase the resistivity

of the crystal structure, thereby improving the magnetic permeability. By introducing bismuth trioxide, Attia Aslam et al. prepared nanoscale powder materials of CoFe_2O_4 and $\text{Sr}_2\text{NiMnFe}_{12}\text{O}_{22}$ [29]. In this context, Bi_2O_3 acted as a scattering center for carriers, hindering carrier hopping between octahedral sites and suppressing the electronic transition mechanism between Fe^{2+} and Fe^{3+} ions. This enhances the resistivity of the composite material and significantly improves its magnetic permeability properties [30,31].

In summary, appropriate Bi^{3+} doping induces lattice distortion, which promotes the magnetic domain transition from a multi-domain state to a single-domain configuration and facilitates cation redistribution between the A-site and B-site. This optimization effectively tailors the magnetic anisotropy and domain wall dynamics, thereby significantly improving the initial permeability of the prepared nanoferrites [32,33].

Therefore, Bi atoms and their corresponding crystal structure played an important role in overcoming Snoek's limit of soft magnetic materials. In this work, a series of CoBiNi alloys with different Bi contents was prepared by the hydrothermal reduction method. These results indicated that the magnetic permeability of CoBiNi alloys could be adjusted by adjusting the doping content. The magnetodielectric properties of the series of cobalt–bismuth–nickel alloys were tested, including the real and imaginary parts of the dielectric constant, as well as the real and imaginary parts of the magnetic permeability; simultaneously, the microwave reflection loss (RL) and effective absorption bandwidth of the alloys were verified. This research provides new insights for the development of high-permeability, low-loss soft magnetic materials.

2. Experimental

2.1. Materials

Nickel acetate tetrahydrate ($\text{C}_4\text{H}_6\text{NiO}_4 \cdot 4\text{H}_2\text{O}$, AR, 98.0%), cobalt acetate tetrahydrate ($\text{C}_4\text{H}_6\text{CoO}_4 \cdot 4\text{H}_2\text{O}$, AR, 99.5%), ethylene glycol ($\text{C}_2\text{H}_6\text{O}_2$, EG, 99%), sodium hydroxide (NaOH, AR, 96%), absolute ethanol ($\text{C}_2\text{H}_5\text{OH}$, $\geq 99.7\%$) and diethylenetriamine ($\text{C}_4\text{H}_{13}\text{N}_3$, DETA, AR) were purchased from Sinopharm Chemical Reagent Co., Ltd. (Shanghai, China). Bismuth nitrate pentahydrate ($\text{Bi}(\text{NO}_3)_3 \cdot 5\text{H}_2\text{O}$, AR, 99.5%) was supplied by Shanghai Qiangshun Chemical Co., Ltd. (Shanghai, China).

2.2. The Preparation Process of $\text{Co}_{20}\text{Bi}_x\text{Ni}_{80-x}$

$\text{Co}_{20}\text{Bi}_x\text{Ni}_{80-x}$ ($x = 1, 2, 5, 10$) alloys were prepared in a solvothermal reduction system as follows: $\text{C}_4\text{H}_6\text{NiO}_4 \cdot 4\text{H}_2\text{O}$, $\text{C}_4\text{H}_6\text{CoO}_4 \cdot 4\text{H}_2\text{O}$ and $\text{Bi}(\text{NO}_3)_3 \cdot 5\text{H}_2\text{O}$ were used as precursors and dissolved in a 40 mL $\text{C}_2\text{H}_6\text{O}_2$ solution; The NaOH concentration was $0.75 \text{ mol} \cdot \text{L}^{-1}$. The precursor concentration was $0.75 \text{ mol} \cdot \text{L}^{-1}$. The $\text{Co}_{20}\text{Bi}_x\text{Ni}_{80-x}$ ($x = 1, 2, 5, 10$) mixture was dissolved by ultrasound at room temperature, and 1 ml of diethylenetriamine (DETA) was added, then transferred into a Teflon-lined stainless steel autoclave (100 mL capacity). The autoclave was sealed and maintained at 200°C for 6 h, then cooled to room temperature. The blackish-gray solid product at the bottom of the reactor was collected and washed twice with anhydrous ethanol, followed by a single wash with deionized water. Finally, the samples were dried in a vacuum oven at 60°C , achieving a dry sample yield of over 85%.

2.3. Characterization

The morphologies and sizes of the $\text{Co}_{20}\text{Bi}_x\text{Ni}_{80-x}$ ($x = 1, 2, 5, 10$) alloys were characterized by a field-emission scanning electron microscope (SEM, Regulus8100, Hitachi, Tokyo, Japan). The distribution of elements of the $\text{Co}_{20}\text{Bi}_x\text{Ni}_{80-x}$ ($x = 1, 2, 5, 10$) alloys was characterized by energy dispersive spectroscopy (EDS, Regulus8100, Hitachi, Tokyo, Japan). The structure of the $\text{Co}_{20}\text{Bi}_x\text{Ni}_{80-x}$ ($x = 1, 2, 5, 10$) alloys was examined by X-ray diffraction (XRD) with a Cu K radiation source at room temperature (D/max-2200/PC, Rigaku, Tokyo,

Japan). The density of the samples was measured by the Archimedes drainage method. The valence state of the element was measured by X-ray photoelectron spectroscopy (Nexsa, Thermo Fisher, Waltham, MA, USA). The magnetic properties of the $\text{Co}_{20}\text{Bi}_x\text{Ni}_{80-x}$ ($x = 1, 2, 5, 10$) alloys were determined with a magnetometer (MPMS-VSM, Quantum Design Company, San Diego, CA, USA). Dielectric constant (ϵ'), dielectric constant (ϵ''), magnetic permeability (μ') and magnetic loss (μ'') values of the samples were measured by a Vector network analyzer (E5080B, Keysight, Santa Rosa, CA, USA). The samples were prepared by homogeneously mixing paraffin wax with composites (mass:ratio = 7:3) and then pressed into toroidal-shaped samples ($\phi_{\text{out}} = 7.0$ mm, $\phi_{\text{in}} = 3.0$ mm).

3. Results and Discussion

The synthesis process of a series of CoBiNi alloys with different Bi contents was illustrated in Figure 1a. As the content increases, both the microstructure and grain size of the cobalt-bismuth-nickel alloy will change. EDS mappings illustrate the existence of Co, Ni and Bi elements in Figure S1. Simultaneously, the difference in Co, Ni and Bi contents needs further analysis during the preparation of the $\text{Co}_{20}\text{Bi}_5\text{Ni}_{75}$ sample. The EDS spectrum is used to test the contents of these elements in the $\text{Co}_{20}\text{Bi}_5\text{Ni}_{75}$ sample, as listed in Table S1. Co contents in three parts of the $\text{Co}_{20}\text{Bi}_5\text{Ni}_{75}$ sample are 7.76%, 15.06% and 5.93%, respectively. Ni contents for those are 87.06%, 83.00% and 93.73%, respectively. Meanwhile, Bi is 5.17%, 1.93% and 0.33%, respectively. Co, Ni and Bi contents at different ports vary greatly, which is consistent with the previous results in Figure S1. SEM images indicate that the $\text{Co}_{20}\text{Ni}_{80}$ alloy presents a chain-like structure (Figure 1b). The length of a single $\text{Co}_{20}\text{Ni}_{80}$ crystal is about 80 μm . In the presence of DETA surfactant, the $\text{Co}_{20}\text{Ni}_{80}$ microspheres self-assemble into a chain-like structure with multiple branches. The preparation conditions of $\text{Co}_{20}\text{Bi}_1\text{Ni}_{79}$, $\text{Co}_{20}\text{Bi}_2\text{Ni}_{78}$, $\text{Co}_{20}\text{Bi}_5\text{Ni}_{75}$, $\text{Co}_{20}\text{Bi}_{10}\text{Ni}_{70}$ and $\text{Co}_{20}\text{Bi}_{80}$ are the same as those of the $\text{Co}_{20}\text{Ni}_{80}$ alloy. A series of CoBiNi alloys all exhibit the chain-like shape. Among them, the diameters of the $\text{Co}_{20}\text{Bi}_1\text{Ni}_{79}$ and $\text{Co}_{20}\text{Bi}_2\text{Ni}_{78}$ alloys are not obviously different from that of $\text{Co}_{20}\text{Ni}_{80}$, whose diameter is below 1 μm , especially in the microspheres' edges. It may be caused by the addition of Bi atoms. With the increase in Bi content, the $\text{Co}_{20}\text{Bi}_5\text{Ni}_{75}$ alloy is assembled into a chain-like structure with a diameter of about 2 μm microspheres in Figure 1e. Its length is about 20 μm . As shown in Figure 1f, the $\text{Co}_{20}\text{Bi}_{10}\text{Ni}_{70}$ alloy is also assembled into a chain-like structure with a diameter of about 1.5 μm and loses the microsphere shape. Meanwhile, there are also some large particles with a rough surface in the $\text{Co}_{20}\text{Bi}_{10}\text{Ni}_{70}$ alloy. As the Bi content increases, the particle size decreases significantly, indicating that Bi atoms inhibit the growth of CoNi grains [26]. The microstructure of $\text{Co}_{20}\text{Bi}_{80}$ alloy is spherical (Figure 1g) with a smooth surface and non-uniform particle size. Meanwhile, the minimum diameter of the observed particles is approximately 500 nm.

According to SEM and EDS elemental mapping results, Bi exhibits a non-uniform distribution in the CoBiNi alloys with local elemental enrichment. Such an inhomogeneous distribution induces local composition fluctuation and heterogeneous interfacial structure, which regulates magnetic domain movement and magnetic exchange coupling, and consequently affects the magnetic permeability and coercivity. To quantitatively reveal the inhibitory effect of Bi on CoNi grain growth, the average crystallite size of all samples was calculated from XRD patterns via the Scherrer equation. With the increase in Bi content, the grain sizes of $\text{Co}_{20}\text{Ni}_{80}$, $\text{Co}_{20}\text{Bi}_1\text{Ni}_{79}$, $\text{Co}_{20}\text{Bi}_2\text{Ni}_{78}$, $\text{Co}_{20}\text{Bi}_5\text{Ni}_{75}$ and $\text{Co}_{20}\text{Bi}_{10}\text{Ni}_{70}$ are 171 Å, 146 Å, 132 Å, 130 Å and 128 Å, respectively. The results showed that the crystallite size gradually decreases with increasing Bi content, confirming that Bi doping can effectively restrain the grain growth of CoNi alloy. The variation trend is consistent with the particle size evolution observed from SEM images, which further verifies the grain refinement

effect of Bi doping. Furthermore, the lattice parameters were calculated according to XRD peak shifts. Due to the larger atomic radius of Bi compared with Co and Ni, Bi incorporation induces obvious lattice distortion and lattice strain, which modulates the electronic structure and serves as an important intrinsic factor for optimizing magnetic properties.

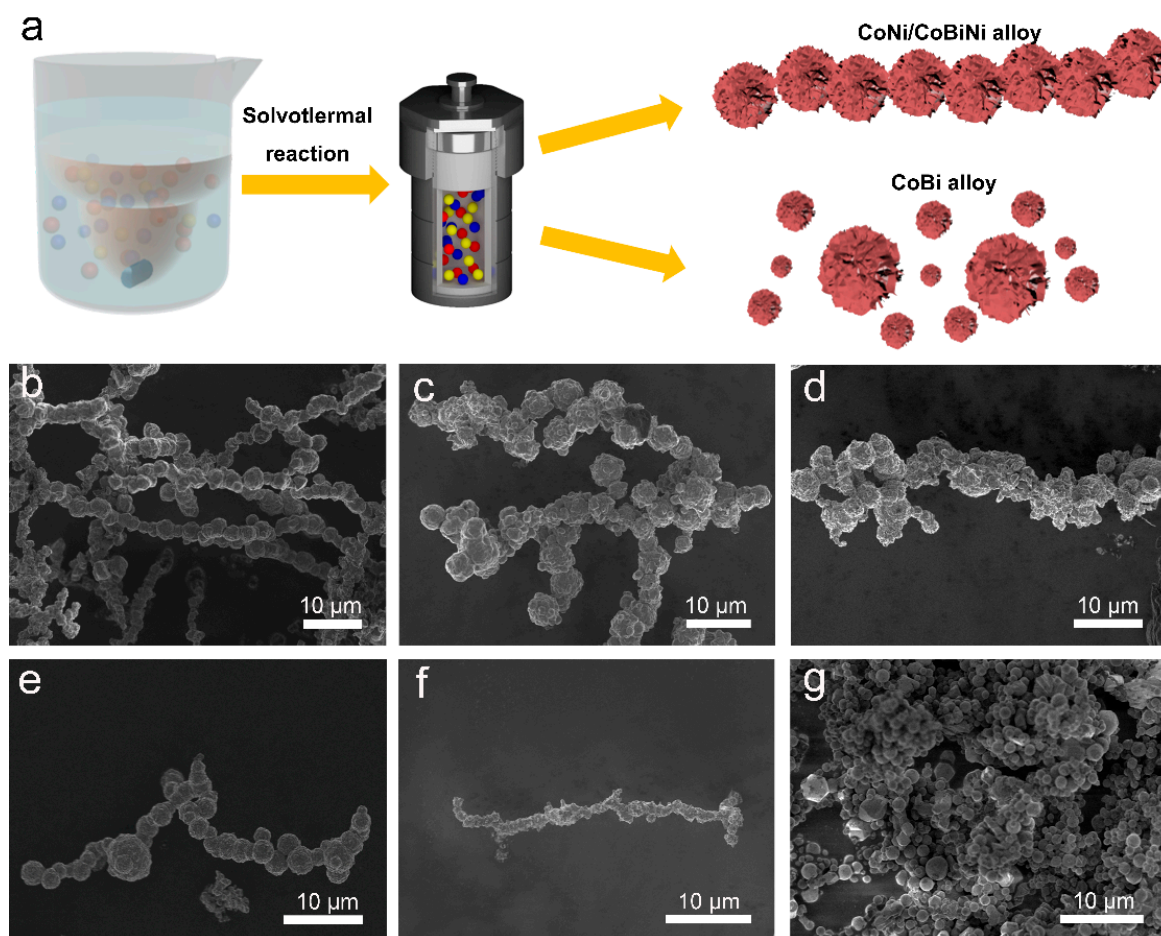


Figure 1. (a) Schematic illustration of the synthetic process of a series of CoBiNi alloys. SEM images of (b) $\text{Co}_{20}\text{Ni}_{80}$, (c) $\text{Co}_{20}\text{Bi}_1\text{Ni}_{79}$, (d) $\text{Co}_{20}\text{Bi}_2\text{Ni}_{78}$, (e) $\text{Co}_{20}\text{Bi}_5\text{Ni}_{75}$, (f) $\text{Co}_{20}\text{Bi}_{10}\text{Ni}_{70}$ and (g) $\text{Co}_{20}\text{Bi}_{80}$.

The crystal structures of $\text{Co}_{20}\text{Ni}_{80}$, $\text{Co}_{20}\text{Bi}_1\text{Ni}_{79}$, $\text{Co}_{20}\text{Bi}_2\text{Ni}_{78}$, $\text{Co}_{20}\text{Bi}_5\text{Ni}_{75}$, $\text{Co}_{20}\text{Bi}_{10}\text{Ni}_{70}$ and $\text{Co}_{20}\text{Bi}_{80}$ alloys are characterized by XRD. As shown in Figure 2a, the diffraction peaks appear at 44.5° , 51.8° and 76.7° , corresponding to the (111), (200) and (220) crystal planes, respectively. It indicates the face-centered cubic (fcc) structure of CoNi alloys. In addition, the diffraction peaks of $\text{Co}_{20}\text{Bi}_{80}$ alloy at 27.1° and 48.7° correspond to the (012) and (202) crystal planes, respectively. Similarly, the diffraction peaks of a series of CoBiNi alloys at 27.1° and 48.7° also correspond to (012) and (202) crystal planes. Meanwhile, the characteristic peaks of $\text{Co}_{20}\text{Ni}_{80}$ alloy are located at 44.5° , 51.8° and 76.7° for (111), (200) and (220) crystal planes. Moreover, the diffraction peak intensities of the CoBiNi alloy series increase with the rise in Bi content. As shown in Figure 2b, the bulk density of CoBiNi alloys also increases with increasing Bi content, and the relative density of alloy samples follows the same trend, verifying that Bi atoms can improve the compactness of the crystal structure [34]. The EDS spectrum indicates that the $\text{Co}_{20}\text{Bi}_5\text{Ni}_{75}$ contains several elements (Figure S1). By detecting the distribution of these elements, the elemental distribution characteristics of the CoBiNi alloy series can be clarified. EDS mapping of Co, Ni and Bi elements is used as tracers to characterize the composition of $\text{Co}_{20}\text{Ni}_{80}$, $\text{Co}_{20}\text{Bi}_5\text{Ni}_{75}$, $\text{Co}_{20}\text{Bi}_{10}\text{Ni}_{70}$ samples. As shown in Figure 2c, only Co and Ni elements are

found in the $\text{Co}_{20}\text{Ni}_{80}$ sample, with no Bi element observed. Comparatively, as shown in Figure 2d,e, Co, Ni and Bi elements are all distributed in the EDS mapping of $\text{Co}_{20}\text{Bi}_5\text{Ni}_{75}$ and $\text{Co}_{20}\text{Bi}_{10}\text{Ni}_{70}$ samples. Combined with XPS analysis, the binding energies of the Bi 4f and $4f_{5/2}$ orbitals were found to be 158.27 eV and 163.58 eV, respectively, indicating that Bi has successfully replaced the magnetic host cations, incorporated into the lattice, and formed a lattice solid solution.

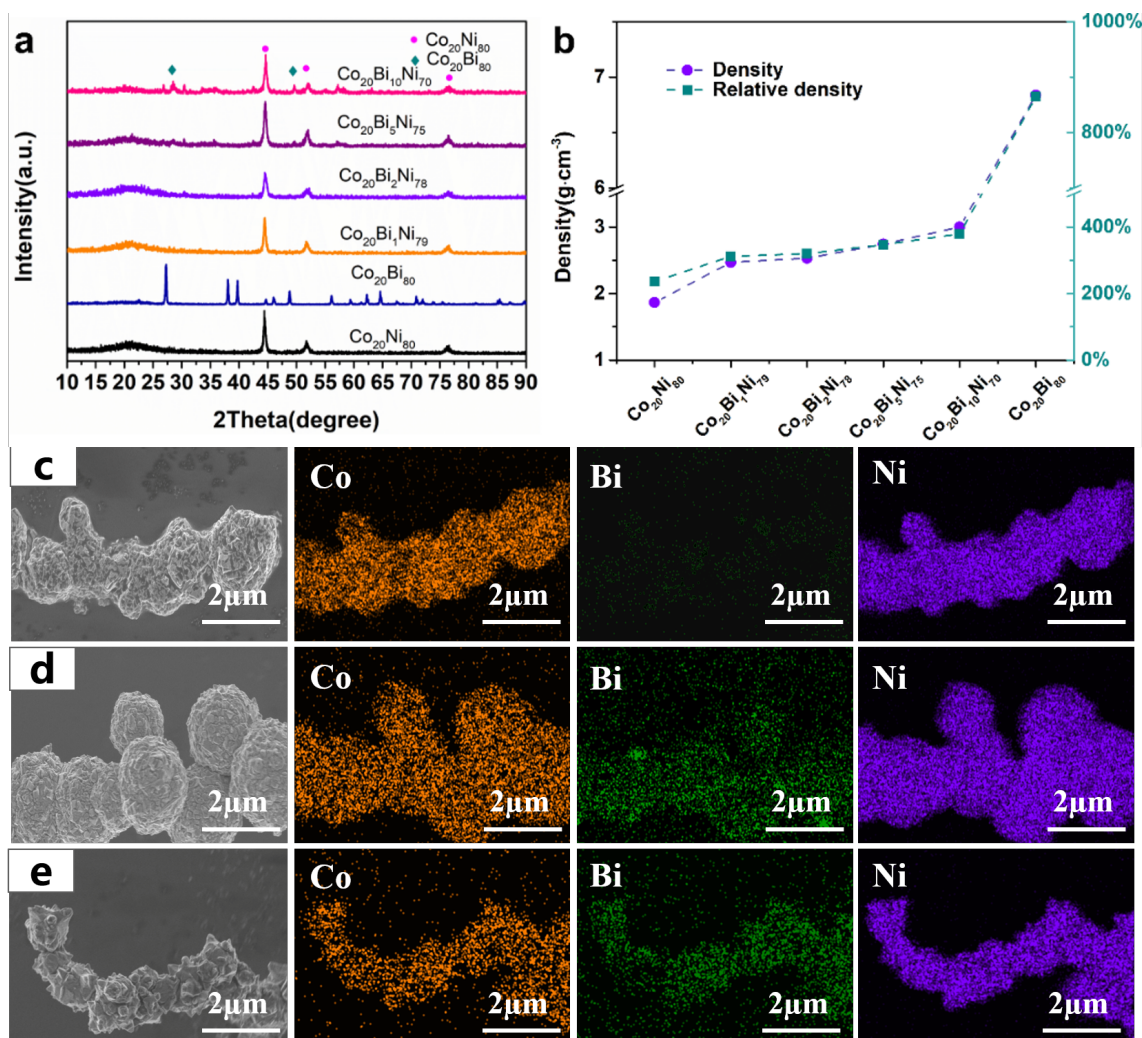


Figure 2. (a) XRD patterns of $\text{Co}_{20}\text{Ni}_{80}$, $\text{Co}_{20}\text{Bi}_{80}$, $\text{Co}_{20}\text{Bi}_1\text{Ni}_{79}$, $\text{Co}_{20}\text{Bi}_2\text{Ni}_{78}$, $\text{Co}_{20}\text{Bi}_5\text{Ni}_{75}$ and $\text{Co}_{20}\text{Bi}_{10}\text{Ni}_{70}$ alloys. (b) Density of $\text{Co}_{20}\text{Ni}_{80}$, $\text{Co}_{20}\text{Bi}_1\text{Ni}_{79}$, $\text{Co}_{20}\text{Bi}_2\text{Ni}_{78}$, $\text{Co}_{20}\text{Bi}_5\text{Ni}_{75}$, $\text{Co}_{20}\text{Bi}_{10}\text{Ni}_{70}$ and $\text{Co}_{20}\text{Bi}_{80}$ alloys. (c–e) SEM image and EDS mapping results of $\text{Co}_{20}\text{Ni}_{80}$, $\text{Co}_{20}\text{Bi}_5\text{Ni}_{75}$ and $\text{Co}_{20}\text{Bi}_{10}\text{Ni}_{70}$ alloys.

The hysteresis loops of a series of CoBiNi alloys measured at 300 K show typical ferromagnetic properties (Figure 3). The saturation magnetizations (M_s) of $\text{Co}_{20}\text{Ni}_{80}$, $\text{Co}_{20}\text{Bi}_1\text{Ni}_{79}$, $\text{Co}_{20}\text{Bi}_2\text{Ni}_{78}$, $\text{Co}_{20}\text{Bi}_5\text{Ni}_{75}$, $\text{Co}_{20}\text{Bi}_{10}\text{Ni}_{70}$ and $\text{Co}_{20}\text{Bi}_{80}$ alloys are 50.81 emu g^{-1} , 68.05 emu g^{-1} , 70.78 emu g^{-1} , 73.11 emu g^{-1} , 56.91 emu g^{-1} and 0.05 emu g^{-1} , respectively. The coercive force (H_c) values of the six samples are 100.59 Oe, 153.80 Oe, 80.98 Oe, 66.28 Oe, 87.36 Oe and 14.47 Oe, while the remanent magnetization (M_r) of the six samples are 4.44 emu g^{-1} , 7.14 emu g^{-1} , 4.80 emu g^{-1} , 2.82 emu g^{-1} , 2.81 emu g^{-1} and 0.002 emu g^{-1} in sequence. Compared with other alloys, the M_s value of the chain-like $\text{Co}_{20}\text{Bi}_5\text{Ni}_{75}$ alloy is the highest due to the existence of multiphases. In addition, the microsphere size of $\text{Co}_{20}\text{Bi}_5\text{Ni}_{75}$ alloy is smaller than that of $\text{Co}_{20}\text{Ni}_{80}$. The interface area of microspheres in $\text{Co}_{20}\text{Bi}_5\text{Ni}_{75}$ alloy increases correspondingly, which strengthens the interfacial exchange

coupling effect between these microspheres, thus effectively enhancing the M_s values [35]. Among all the samples, $\text{Co}_{20}\text{Bi}_1\text{Ni}_{79}$ alloy has the largest coercivity, followed by $\text{Co}_{20}\text{Ni}_{80}$ alloy, due to its large shape anisotropy and large particle size [20]. However, $\text{Co}_{20}\text{Bi}_5\text{Ni}_{75}$ alloy has the lowest coercivity of 66.28 Oe, which is attributed to its small particle size [9]. The residual magnetization of all five main alloys is relatively low, indicating that these materials are typical soft magnetic materials.

Figure 4 presents the four magnetic and dielectric properties of $\text{Co}_{20}\text{Ni}_{80}$, $\text{Co}_{20}\text{Bi}_1\text{Ni}_{79}$, $\text{Co}_{20}\text{Bi}_2\text{Ni}_{78}$, $\text{Co}_{20}\text{Bi}_5\text{Ni}_{75}$ and $\text{Co}_{20}\text{Bi}_{10}\text{Ni}_{70}$ alloys. Figure 4a shows the real permittivity ϵ' of five alloy materials. It is obvious that the ϵ' of $\text{Co}_{20}\text{Ni}_{80}$ decreases at 1.00–18.00 GHz, which is mainly caused by the motion hysteresis of charged ions under an alternating electric field, resulting in the magnetic moment relaxation phenomenon. The ϵ' of materials reflects their polarization capability [8,36], and a higher ϵ' corresponds to a higher polarization degree. After the doping of Bi atoms, the ϵ' values of $\text{Co}_{20}\text{Bi}_1\text{Ni}_{79}$, $\text{Co}_{20}\text{Bi}_5\text{Ni}_{75}$, $\text{Co}_{20}\text{Bi}_2\text{Ni}_{78}$ and $\text{Co}_{20}\text{Bi}_{10}\text{Ni}_{70}$ are significantly lower than those of $\text{Co}_{20}\text{Ni}_{80}$ at 1.00–18.00 GHz. The ϵ' value of $\text{Co}_{20}\text{Bi}_5\text{Ni}_{75}$ also decreases with increasing frequency, and remains stable at approximately 10.26 in the range of 9.56–18.00 GHz. This phenomenon is attributed to the incorporation of Bi atoms, which increases the material's resistivity and reduces the electron hopping probability in the crystal structure. As shown in Figure 4b, the imaginary permittivity (ϵ'') of $\text{Co}_{20}\text{Ni}_{80}$ reaches 15.98 at 4.53 GHz, and exhibits obvious fluctuations across the entire tested frequency band of 1.00–18.00 GHz. After the addition of the Bi element, the ϵ'' value of $\text{Co}_{20}\text{Bi}_1\text{Ni}_{79}$ and $\text{Co}_{20}\text{Bi}_2\text{Ni}_{78}$ remains stable. In the range of 1.00–18.00 GHz, the ϵ'' values of $\text{Co}_{20}\text{Bi}_5\text{Ni}_{75}$ alloy fluctuate slightly from 23.04 to 0.56, while obvious sharp fluctuations appear at 1.00–2.00 GHz and 8.00–10.00 GHz, respectively. The ϵ'' peak value of $\text{Co}_{20}\text{Bi}_{10}\text{Ni}_{70}$ alloy is approximately 5.80 at 11.63 GHz.

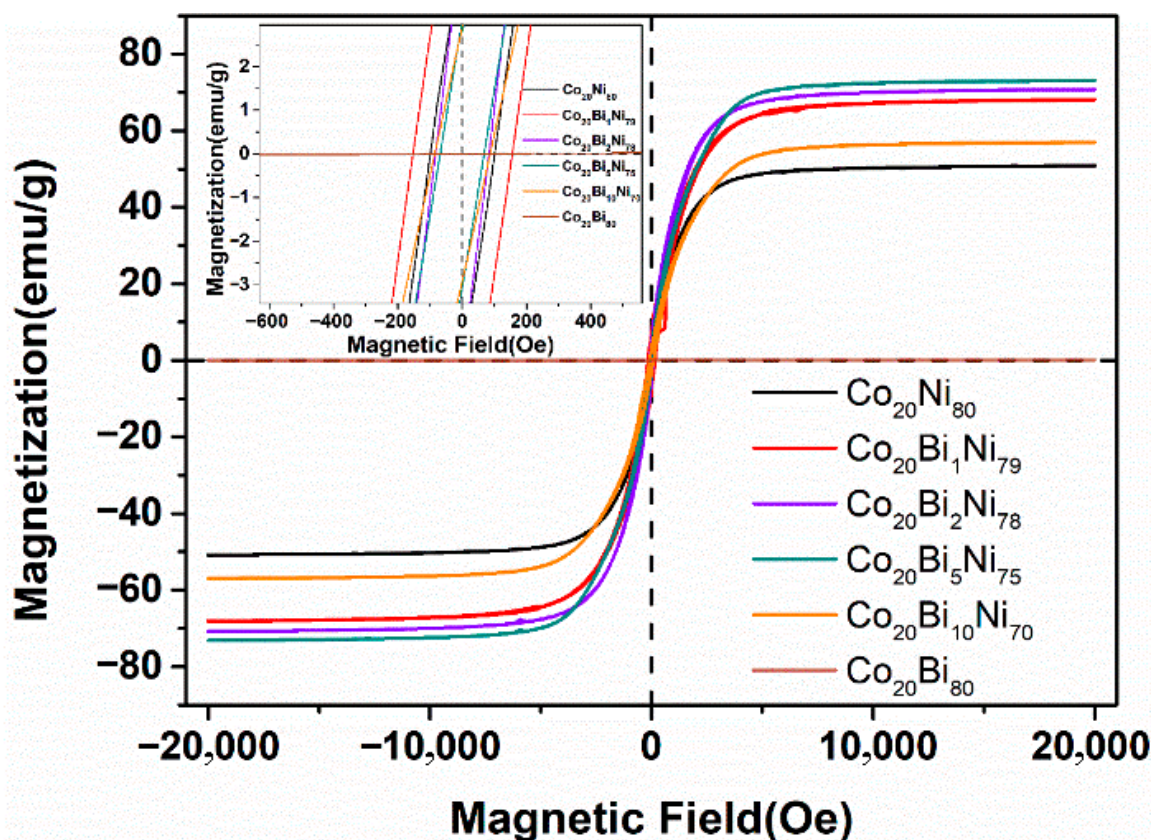


Figure 3. Hysteresis loops of $\text{Co}_{20}\text{Ni}_{80}$, $\text{Co}_{20}\text{Bi}_1\text{Ni}_{79}$, $\text{Co}_{20}\text{Bi}_2\text{Ni}_{78}$, $\text{Co}_{20}\text{Bi}_5\text{Ni}_{75}$, $\text{Co}_{20}\text{Bi}_{10}\text{Ni}_{70}$ and $\text{Co}_{20}\text{Bi}_{80}$ alloys measured at 300 K.

To further clarify the magnetic loss mechanism, the eddy current loss coefficient $C_0 = \mu''(\mu')^{-2}f^{-1}$ was calculated, and the results are presented in Figure 5. Theoretically, if eddy current loss dominates the magnetic loss, C_0 should remain constant over the measured frequency range. However, all samples in this work show obvious frequency-dependent C_0 values, demonstrating that eddy current loss is not the dominant magnetic loss mechanism within 1–14 GHz. Only $\text{Co}_{20}\text{Ni}_{80}$ exhibits evident C_0 fluctuation above 14 GHz, indicating a minor contribution of eddy current loss at high frequencies. With the increase in Bi content, the C_0 value decreases gradually and becomes more stable. Combined with SEM, EDS, XRD and XPS results, the evolution of electromagnetic properties is closely related to microstructure characteristics: Bi doping refines grain size, inhibits grain growth, and maintains the chain-like self-assembled morphology; the non-uniform distribution of Bi induces local composition fluctuation and increases electrical resistivity, which effectively suppresses long-range electron migration and eddy current effect. Therefore, magnetic loss is mainly dominated by natural resonance and exchange resonance, which is intrinsically determined by the chain-like structure, grain size effect and inhomogeneous Bi distribution of CoBiNi alloys.

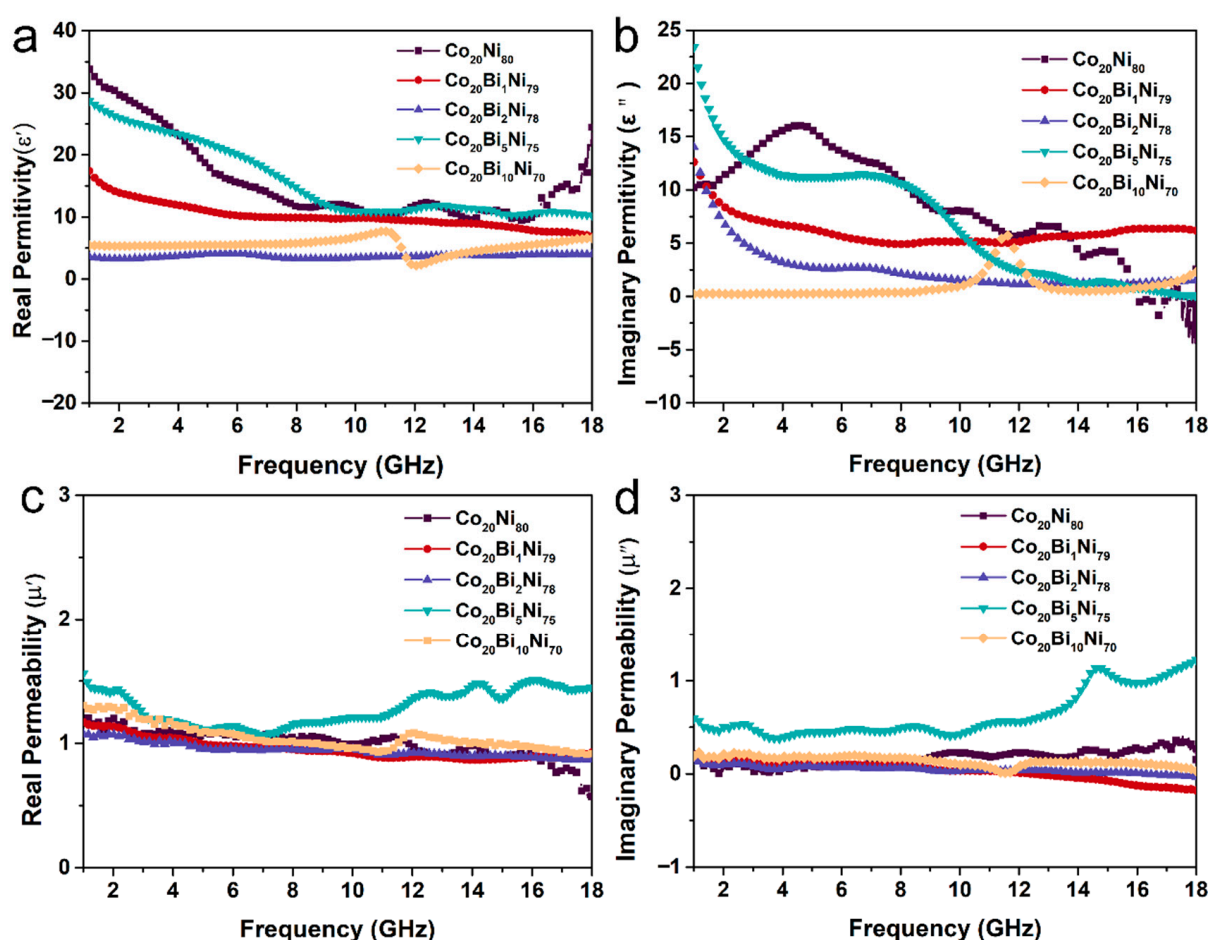


Figure 4. (a) ϵ' of a series of CoBiNi alloys. (b) ϵ'' of a series of CoBiNi alloys. (c) μ' of a series of CoBiNi alloys. (d) μ'' of a series of CoBiNi alloys.

Figure 6c,d separately present the μ' and μ'' curves of $\text{Co}_{20}\text{Ni}_{80}$, $\text{Co}_{20}\text{Bi}_1\text{Ni}_{79}$, $\text{Co}_{20}\text{Bi}_2\text{Ni}_{78}$, $\text{Co}_{20}\text{Bi}_5\text{Ni}_{75}$ and $\text{Co}_{20}\text{Bi}_{10}\text{Ni}_{70}$ alloys. The μ' value of $\text{Co}_{20}\text{Ni}_{80}$ remains about 1.17 within the frequency range of 1.00–18.00 GHz. When the Bi doping content is increased to 1% and 2%, the μ' values of $\text{Co}_{20}\text{Bi}_1\text{Ni}_{79}$ and $\text{Co}_{20}\text{Bi}_2\text{Ni}_{78}$ are still maintained at around 1.17 throughout the tested frequency band of 1.00–18.00 GHz, respectively. As

the Bi content increases to 5%, the μ' values of $\text{Co}_{20}\text{Bi}_5\text{Ni}_{75}$ fluctuate around 1.50 in the range of 14.00–18.00 GHz, which may be attributed to the combined effect of eddy current loss, anomalous loss and enhanced polarization ability caused by the incorporation of Bi atoms [36]. The μ'' values of $\text{Co}_{20}\text{Ni}_{80}$, $\text{Co}_{20}\text{Bi}_1\text{Ni}_{79}$ and $\text{Co}_{20}\text{Bi}_2\text{Ni}_{78}$ basically fluctuate within the scope of 0–0.30 at 1.00–18.00 GHz. During the same range, the μ' values show a continuous decreasing trend, which is caused by eddy current losses and abnormal losses at high frequency. The μ'' value of the $\text{Co}_{20}\text{Bi}_5\text{Ni}_{75}$ alloy fluctuates around 0.50 at the frequency range of 1.00–12.00 GHz. Similarly, the μ'' value increases in the range of 12.00–18.00 GHz and finally reaches 1.21 at 18.00 GHz.

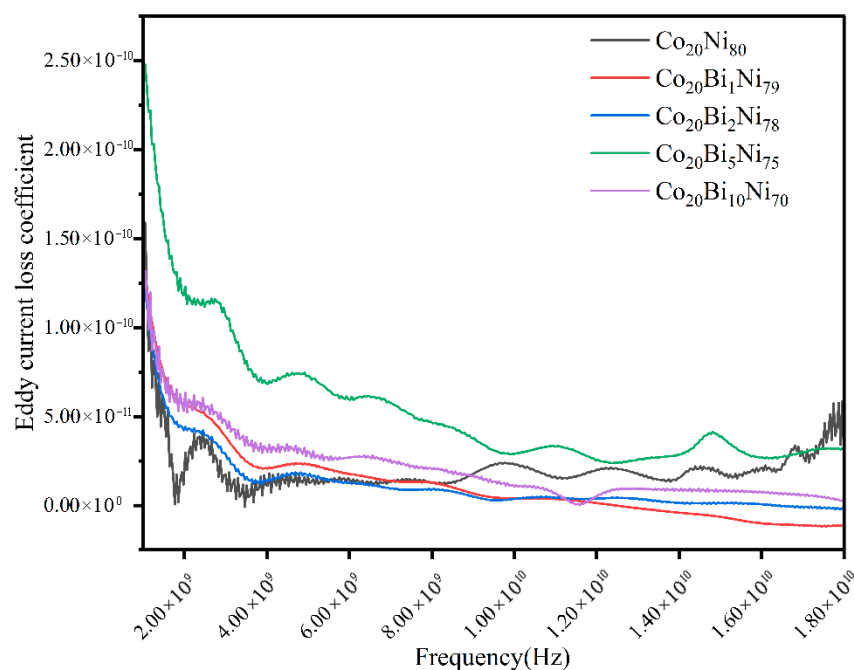


Figure 5. Frequency dependence (1–18 GHz) of the eddy current loss coefficient $C_0 = \mu''(\mu')^{-2}f^{-1}$ for $\text{Co}_{20}\text{Ni}_x\text{Bi}_y$ samples with different Bi contents.

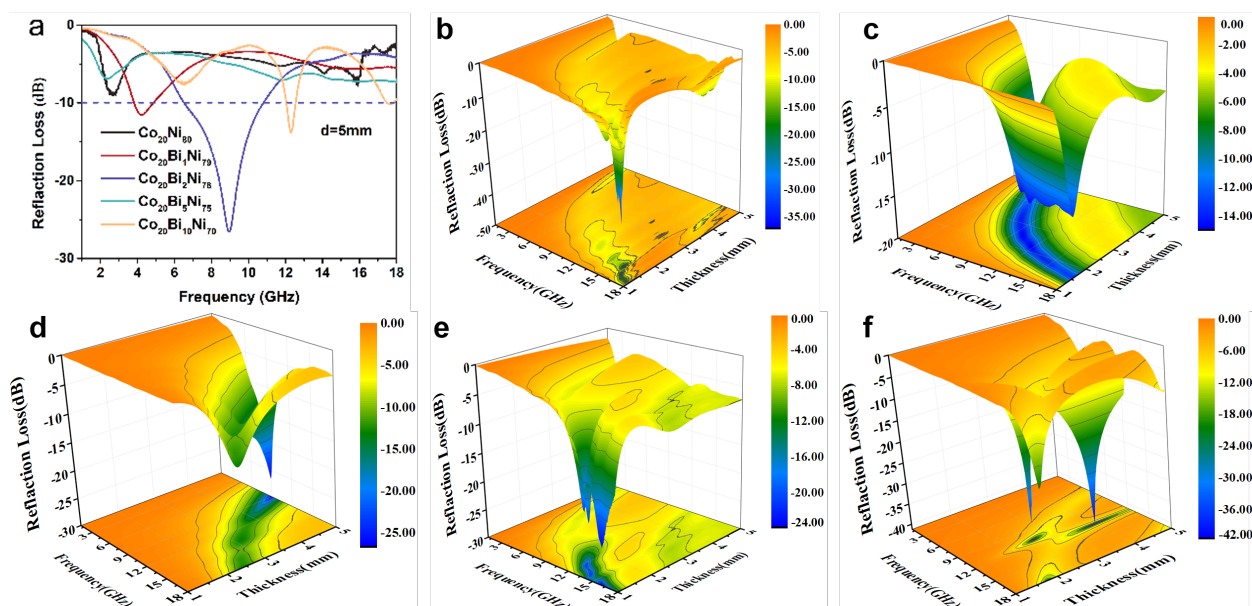


Figure 6. (a) RL curves of five samples at the same thickness of 1mm; 3D plots of RL for (b) $\text{Co}_{20}\text{Ni}_{80}$, (c) $\text{Co}_{20}\text{Bi}_1\text{Ni}_{79}$, (d) $\text{Co}_{20}\text{Bi}_2\text{Ni}_{78}$, (e) $\text{Co}_{20}\text{Bi}_5\text{Ni}_{75}$ and (f) $\text{Co}_{20}\text{Bi}_{10}\text{Ni}_{70}$.

According to the above electromagnetic parameters, the RL values of the five alloys were calculated, as shown in Figure 6a. The $\text{Co}_{20}\text{Ni}_{80}$ sample exhibits no microwave absorption capacity at a thickness of 1 mm. The RL value of the $\text{Co}_{20}\text{Bi}_5\text{Ni}_{75}$ sample exceeds -6.89 dB at 2.34 GHz under a thickness of 5 mm. Therefore, Bi atoms can effectively improve the signal transmission efficiency for 5G communication. To further illustrate the effect of Bi atoms on microwave absorption performance, the correlation between absorption performance and frequency for alloy samples with different thicknesses was systematically calculated. Figure 6b–f show the 3D RL curves of CoNi , $\text{Co}_{20}\text{Bi}_1\text{Ni}_{79}$, $\text{Co}_{20}\text{Bi}_2\text{Ni}_{78}$, $\text{Co}_{20}\text{Bi}_5\text{Ni}_{75}$ and $\text{Co}_{20}\text{Bi}_{10}\text{Ni}_{70}$ samples with different thicknesses. $\text{Co}_{20}\text{Ni}_{80}$ has a maximum RL value of 36.98 dB at 1.5 mm thickness due to its high real permittivity. As shown in Figure 6c, the RL values of $\text{Co}_{20}\text{Bi}_1\text{Ni}_{79}$ alloy are -14.82 dB at 11.97 GHz with a thickness of 2.15 mm. The RL values of $\text{Co}_{20}\text{Bi}_2\text{Ni}_{78}$ alloy are -26.47 dB at 8.90 GHz with a thickness of 2.15 mm (Figure 6d). Significantly, with the increase in Bi content, the $\text{Co}_{20}\text{Bi}_5\text{Ni}_{75}$ alloy delivers an optimal RL value of -23.75 dB at a thickness of 1.00 mm, and its corresponding effective absorption bandwidth reaches 5 GHz (Figure 6f). Similarly, the RL value of $\text{Co}_{20}\text{Bi}_{10}\text{Ni}_{70}$ alloy is -36.88 dB at 10.86 GHz under a thickness of 2.60 mm. Combined with the magnetic properties and microwave absorption performance, moderate doping of Bi atoms is beneficial to improve the microwave absorption properties of the soft magnetic materials.

To further clarify the regulatory effect of Bi doping, Figure 7 displays the full XPS survey and high-resolution spectra of the $\text{Co}_{20}\text{Bi}_5\text{Ni}_{75}$ alloy. Figure 7a shows the three characteristic peaks of the C1s spectrum, which correspond to C-C (284.91 eV), C-O-C (286.03 eV) and O-C=O (288.86 eV), respectively. The high-resolution Co 2p spectrum confirms the coexistence of metallic Co and Co^{2+} ions. The peaks located at 778.75 eV and 793.33 eV are assigned to $\text{Co } 2p^{3/2}$ and $\text{Co } 2p^{1/2}$, respectively. These results verify that the main valence state of Co is metallic Co, while the peaks at 782.65 eV and 798.25 eV belong to Co^{2+} ions. According to the Ni 2p spectrum in Figure 7c, the peaks at 853.85 and 871.20 eV are assigned to the characteristic peaks of Ni^0 for $\text{Ni } 2p^{3/4}$ and $\text{Ni } 2p^{1/2}$, respectively. In addition, the peaks at 859.30 and 875.02 eV indicate the presence of Ni^{2+} ions. In Figure 7d, the Bi 4f spectrum contains two peaks located at 158.27 eV for $\text{Bi}^0 4f^{7/2}$ and 163.58 eV for $\text{Bi}^0 4f^{5/2}$. Furthermore, there are two more peaks at 160.45 eV and 165.81 eV, which correspond to $\text{Bi}^{3+} 4f^{7/2}$ and $\text{Bi}^{3+} 4f^{5/2}$, respectively. These XPS results further demonstrate that the $\text{Co}_{20}\text{Bi}_5\text{Ni}_{75}$ sample contains abundant Co^{2+} , Ni^{2+} and Bi^{3+} ions besides the corresponding metallic elemental valence states. This result indicates that Bi exists in two coexisting chemical states in the as-prepared alloy; part of Bi is present in the form of metallic clusters, while the rest exists in the oxidized state as Bi^{3+} . The Bi^{3+} species may originate from surface oxidation during material preparation or air exposure, and may also segregate at the grain boundaries or surface regions of the alloy. Furthermore, it should be noted that the XPS analysis was only performed on representative samples. For other compositions in the series (e.g., samples with different Bi contents or Co/Ni ratios), the chemical state and relative proportion of Bi may vary with composition and preparation conditions, which constitutes one limitation of this study. The movement of charge carriers among them causes interface polarization and reduces magnetic properties. Based on the available XPS results, we speculate that Bi^{3+} may act as carrier scattering centers to locally increase the resistivity of the material, thereby exerting an indirect influence on magnetic permeability.

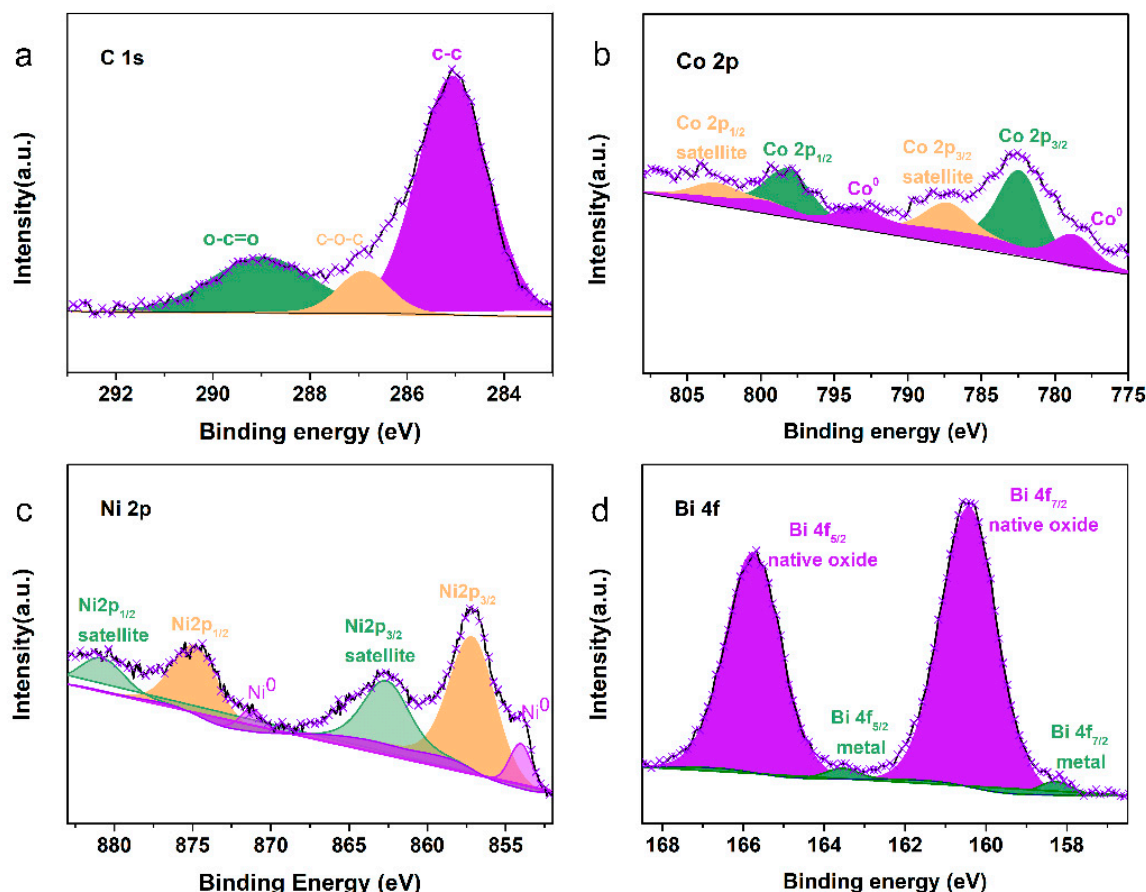


Figure 7. (a) C 1s XPS spectrum of $\text{Co}_{20}\text{Bi}_5\text{Ni}_{75}$; (b) Co 2p XPS spectrum of $\text{Co}_{20}\text{Bi}_5\text{Ni}_{75}$; (c) Ni 2p XPS spectrum of $\text{Co}_{20}\text{Bi}_5\text{Ni}_{75}$; (d) Bi 4f XPS spectrum of $\text{Co}_{20}\text{Bi}_5\text{Ni}_{75}$.

4. Conclusions

In this work, a series of CoBiNi alloys were successfully fabricated via a simple structural template combined with a hydrothermal reaction method. The effect and mechanism of Bi doping on regulating the magnetic permeability of the materials were revealed. Adjusting the Bi content can effectively modulate the compactness and magnetic properties of the alloys. Among them, the $\text{Co}_{20}\text{Bi}_5\text{Ni}_{75}$ sample exhibits the optimal magnetic permeability, reaching 1.5 at 16.56 GHz. Furthermore, microwave reflection loss (RL) and saturation magnetization characterizations demonstrate that $\text{Co}_{20}\text{Bi}_5\text{Ni}_{75}$ possesses excellent microwave absorption capacity and high saturation magnetization. With a sample thickness of 5 mm, its RL value remains higher than -6.89 dB within the frequency range of 1.00–18.00 GHz. Based on the XPS results, it is preliminarily speculated that the superior magnetic permeability of CoBiNi alloys is attributed to Bi content, which hinders the migration of charge carriers and increases the material resistivity, thereby improving the magnetic permeability. These provide a reference for the design and development of functional materials with tunable magnetic permeability.

Supplementary Materials: The following supporting information can be downloaded at: <https://www.mdpi.com/article/10.3390/met16050539/s1>, Table S1. EDS data of $\text{Co}_{20}\text{Bi}_5\text{Ni}_{75}$ sample; Figure S1. (a) SEM images and EDS mapping results of $\text{Co}_{20}\text{Bi}_5\text{Ni}_{75}$; Figure S2. (a) Real permittivity (ϵ') of $\text{Co}_{20}\text{Bi}_{80}$ alloy. (b) Imaginary permittivity (ϵ'') of $\text{Co}_{20}\text{Bi}_{80}$ alloy. (c) Real permeability (μ') of $\text{Co}_{20}\text{Bi}_{80}$ alloy. (d) Imaginary permeability (μ'') of $\text{Co}_{20}\text{Bi}_{80}$ alloy; Figure S3. (a) RL curves of $\text{Co}_{20}\text{Bi}_{80}$ at the same thickness of 5 mm; Figure S4. 3D representations of reflection loss of $\text{Co}_{20}\text{Bi}_{80}$.

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