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The effects of field annealing on the magnetic properties of FeSiB amorphous powder cores

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ABSTRACT

The effects of transverse magnetic field annealing on the magnetic properties of $Fe_{78}Si_9B_{13}$ amorphous powder cores were investigated. The cores annealed at 400 °C under an external transverse magnetic field of 0.5 T show an enhanced and stable effective permeability of 90 in the high frequency range up to 2 MHz. Meanwhile, the permeability was improved by about 5% and the core loss was decreased by about 10% compared to the core with zero field annealing, which is attributed to the relief of internal stress and the variation of domain structure. It also shows superior DC bias property with a "percent permeability" of 70% at an external field of 100 Oe. Transverse magnetic field annealing is an effective way to improve the soft magnetic properties of the amorphous powder cores. The improvement of soft magnetic properties of the Fe-based amorphous powder cores is encouraging for future applications as functional materials.

1. Introduction

Soft magnetic composites (SMCs) with high saturation flux density, stable permeability and low core loss at high frequency are of interest for applications in various electronic devices. After decades of development, different material systems including Fe-Si, Fe-Si-Al, Fe-Ni, Fe-Ni-Mo, Fe-Si-B and Fe-Cu-Nb-Si-B were studied as SMCs [1–6]. Among these systems, the Fe-based Fe-Si-B amorphous alloy exhibits good properties as soft magnetic materials being the combination of advantages of conventional powder core, such as high saturation induction, high permeability, low coercive field and low core loss, and can be widely used in electronic devices of magnetic shielding, transformer, and choking coil [7–10]. There have lots of researches for the effect of different parameters on the magnetic properties of the amorphous powder cores, and found that the magnetic properties of the final cores can be improved by adjusting the amorphous powder size, the characteristics and contents of the binding materials, the consolidation conditions, the shape of the core and the annealing temperature and time [11–14]. Besides these traditional methods, in order to fulfill the demands of high frequency, miniaturization and large current for electronic components, it is inevitable to develop other new methods to improve the magnetic properties of the amorphous powder cores.

Magnetic field annealing is a thermal processing under the presence of external magnetic field [15]. K. Suzuki and G. Herzer researched the effect of field annealing on Magnetic field induced anisotropies and exchange softening in Fe-rich nanocrystalline soft magnetic alloys [16]. A. Kolano-Burian discussed Magnetic domain structure and transverse induced magnetic anisotropy in CoFe-CuNbSiB alloys [17]. V. Procházka and Reisho Onodera studied the effect of magnetic field on crystalline about Fe-based amorphous and nanocrystalline alloys [18,19]. From all these works, it is confirmed that for soft magnetic alloys, magnetic field annealing can effectively improve the soft magnetic properties through eliminating the residual stress and improving the magnetic domain structure [20,21]. However, there is seldom research about the effect of magnetic field annealing on the soft magnetic properties of amorphous powder cores.

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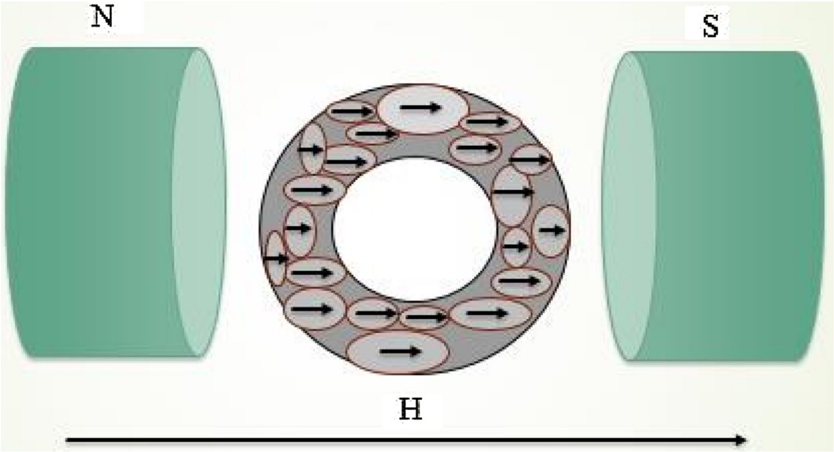

Fig. 1. The schematic diagram of the diection of magnetic field annealing.

In the present work, in order to further improve the soft magnetic properties of the $Fe_{78}Si_9B_{13}$ amorphous powder cores, we introduced a transverse magnetic field of 0.5 T during the annealing process of the cores. And the effects of transverse magnetic field annealing on the magnetic properties of the cores were investigated. It is found that the transverse magnetic field annealing is beneficial to improve the permeability and reduce the core loss of the powder cores. The mechanisms of the phenomenon were also discussed.

2. Experimental

Fe-Si-B amorphous powder was derived from crushing $Fe_{78}Si_9B_{13}$ amorphous ribbons by ball milling as the original powder. The powder was sieved into 50–150 μm to fabricate the powder cores by cold pressing. The powder was passivated by 8 wt.% phosphating solution and uniformly mixed with 4 wt.% of organic binders (2 wt.% epoxy resin and 2 wt.% polyamide resin). The phosphating solution and binders were dissolved with the help of acetone and ethylalcohol as co-solvents. The composite powder was then dried for 30 minutes in an electric thermostatic drying oven. The toroid-shaped amorphous powder cores with dimensions of 20.3 mm in outer diameter, 12.7 mm in inner diameter and 6.35 mm in thickness (Φ 20.3 × Φ 12.7 × t 6.35 mm) were prepared by cold pressing under a pressure of 1.8 GPa at room temperature. Then the compacted cores were annealed at a transverse magnetic field of 0.5 T in 350, 400 and 450 °C for 30 min to improve the soft magnetic properties. The direction of magnetic field and the location of the amorphous powder cores were shown in Fig. 1. The magnetic lines were shown as the symbol of arrows. The direction of the magnetic field is perpendicular the long axis of the powder core. Transverse magnetic field annealing make the easy magnetization axis have a tendency to along the direction of magnetic lines. The phase structure of the $Fe_{78}Si_9B_{13}$ amorphous powder was analyzed by X-ray diffraction (XRD) with Cu *K*α radiation. The original morphology of the amorphous powders with different sizes was observed by scanning election microscopy (SEM). The permeability of the powder cores was calculated from core inductance measured by Agilent 4294A Impedance Analyzer. The DC bias field performance was measured by Agilent 4284 A LCR meter. The magnetic core loss was measured by an AC B–H loop analyzer.

3. Results and discussion

The XRD patterns of the $Fe_{78}Si_9B_{13}$ amorphous powder were shown in Fig. 2. It was found that only a diffuse halo pattern typical for an amorphous phase is seen within the range of $2\theta = 45°$, and no detectable sharp bragg peaks of a crystalline phase is observed, indicating that the powder with a fully amorphous phase is derived from crushing the corresponding rapidly quenched ribbons. The morphology of the sieved powders was examined by SEM and shown in Fig. 2 as a topmost inset. It was observed that the powders had shape like flakes with sharp edges and corners, which means that the power is difficult to insulate completely. So in order to decrease the core loss of the final amorphous powder cores, more passivator and insulator materials are needed to make the amorphous powder separated from each other. The thickness was much smaller than dimensions in two remaining directions. It can be seen that the sieved particles have a relatively uniform size around 100 μm, which was suitable for forming consolidated powder cores with high density.

Fig. 3 shows frequency dependence of the permeability for the annealed $Fe_{78}Si_9B_{13}$ amorphous powder cores. It can be seen that no obvious decreasing in permeability can be found for all the amorphous powder cores even with increasing frequency up to ~2 MHz because of completely insulation between the amorphous powders. When the annealing temperature increases from 350 to 400 °C, the permeability of the cores at 100 kHz increases from 75 to 85, further increasing the annealing temperature to 450 °C, the permeability decreases to only 60. In general, amorphous and nanocrystalline alloys have their optimum annealing temperatures or temperatures range to eliminate the residual stress and get the best magnetic properties, exorbitant temperature will make

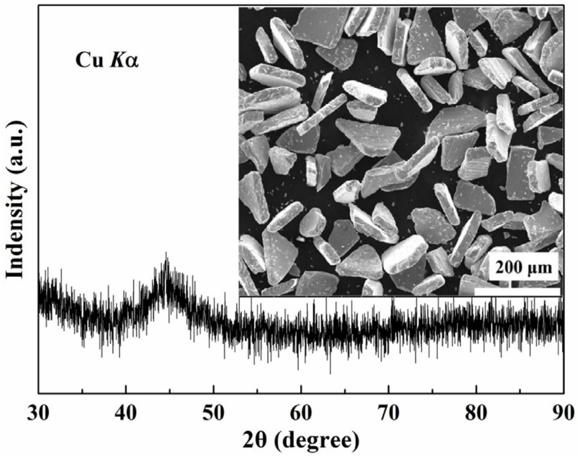

Fig. 2. XRD pattern and SEM image of the $Fe_{78}Si_9B_{13}$ amorphous powders.

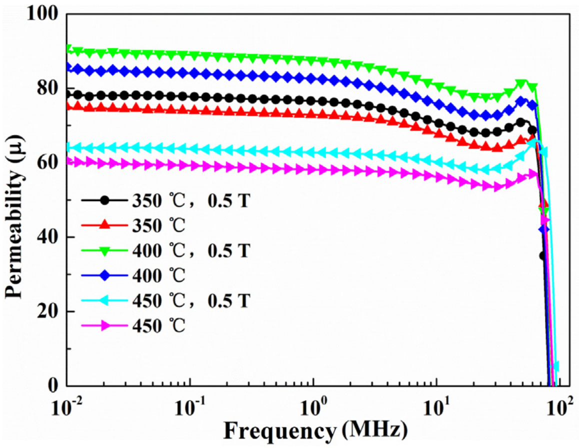

Fig. 3. Frequency dependence of the permeability for the annealed $Fe_{78}Si_9B_{13}$ amorphous powder cores annealed at different conditions.

materials generate new phase or phase grows bigger, which will worsen the soft magnetic properties. For the powder cores annealed at 350 °C, the residual stress failed to be completely removed, so the permeability keeps to a lower level. When the annealing temperature is 400 °C, the annealing process can provide a high-uniformity film, as well as a low volume fraction of defects, and reduces the distortion within the powders, so the permeability of the amorphous powder cores increases. The later decreases can be explained by the precipitation of some hard magnetic crystallites, which serve as pinning centers retarding movement of the domain walls and deteriorated the soft magnetic properties when annealed at 450 °C. As the permeability reaches to a maximum value when annealed at 400 °C, we can conclude that the optimum annealing temperature is 400 °C for the $Fe_{78}Si_9B_{13}$ amorphous powder cores. It can also be seen that for all the samples, when an external magnetic field of 0.5 T was applied during annealing, the permeability was improved by about 5% compared to the core with zero field annealing. This trend can be more clearly seen in Fig. 4. At frequency of 100 kHz, the permeability of the core annealed at 0.5 T increases from 75 to 79 at 350 °C, 85 to 90 at 400 °C, and 60 to 63.5 at 450 °C. As we all known, higher permeability represent better soft magnetic properties. Conventional vacuum annealing can just release the stress in amorphous and nanocrystalline alloys, and cannot change the domain structure, but field annealing can change the domain structure [22]. So the enhancement of permeability annealed in magnetic field of 0.5 T can be attributed to the eliminating of residual stress and improving of the magnetic domain structure [20]. As mentioned above, the optimum annealing temperature is 400 °C, so the $Fe_{78}Si_9B_{13}$ amorphous powder cores annealed at 400 °C with a transverse magnetic field of 0.5 T can get the highest permeability of 90.

Core loss (P_{cv}) is an important parameter for soft magnetic devices. Fig. 5 shows the frequency dependence of the core loss of $Fe_{78}Si_9B_{13}$ amorphous powder cores under the maximum magnetic flux density (B_m) of 100 mT. The P_{cv} decreases from 312 to 260 W/kg when the annealing temperature increases from 350 to 400 °C, further increasing the annealing temperature to 450 °C, the P_{cv} rapidly increases to 1080 W/kg. As we know, P_{cv} is consist of hysteresis loss (P_h), eddy current loss (P_e) and residual loss (P_r), which can be expressed by Eq. (1) [23,24]:

$$P_{cv} = P_h + P_e + P_r \tag{1}$$

P_r is a combination of relaxation and resonant losses. It is only important at very low induction levels and very high frequencies and can be ignored in power applications [25]. So P_{cv} of a magnetic device is the sum of P_e and P_h. The P_h is mainly influenced by the coercive force [26], which is a structure sensitive soft magnetic property and depends on the microstrain accompanied by structural defects. The microstrain and the internal stress can be eliminated effectively under higher temperature annealing, resulting the decrease of the coercive force and the P_h, thus the total P_{cv} drops with the annealing temperature increasing from 350 to 400 °C. Further increasing the annealing temperature to 450 °C, some hard magnetic crystallites may precipitated in the sample, resulted in the increasing of the coercive force and P_h. Meanwhile, the insulation layer begins to decompose rapidly and the P_e between amorphous powders also increases, so the total P_{cv} increases quickly. It can also be seen that field annealing can reduce the P_{cv} of the amorphous cores comparing to vacuum annealing. The field annealing around the curie temperature is easy to obtain an order of atomic arrangement and form a ordered structure. It changes the shape of hysteresis loop and decreases the P_h, resulting in the further decreases of the P_{cv}. As a result, when the amorphous powder core annealed at 400 °C and under a transverse magnetic field of 0.5 T, it exhibits the lowest P_{cv} of 235 W/kg at 100 kHz.

Fig. 5 also shows that the core loss increases monotonously and greatly with increasing of frequency for all the samples. As we know, P_h and P_e can be expressed as Eqs. (2) and (3) [27]:

$$P_h = K_H B^3 f \tag{2}$$

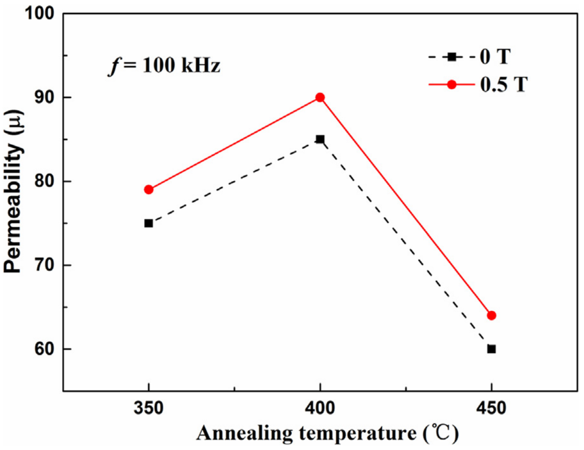

Fig. 4. The permeability changes of the amorphous powder cores as a function of annealing temperature at 100 kHz.

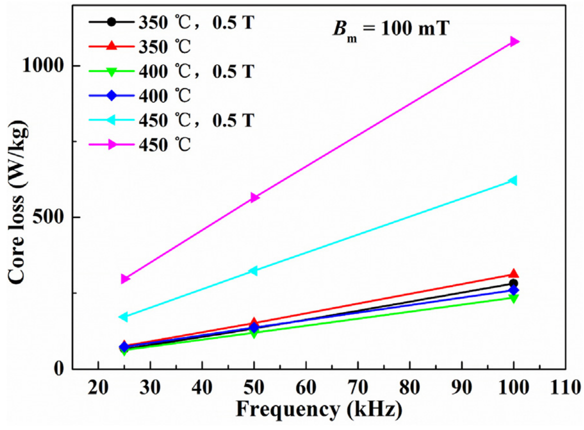

Fig. 5. Frequency dependence of the core loss for the $Fe_{78}Si_9B_{13}$ amorphous powder cores annealed at different conditions.

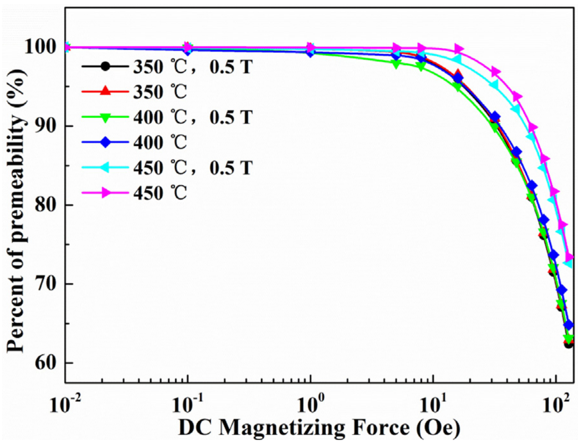

Fig. 6. The dependence of permeability change on bias field at 100 kHz for the $Fe_{78}Si_9B_{13}$ amorphous powder cores annealed at different conditions.

$$P_e = \frac{K_E B^2 f^2 d^2}{\rho} \quad (3)$$

where K_H and K_E are proportionality constants, B is the induction level, f is the frequency of the varying magnetic field, d is effective dimension, and ρ is the electrical resistivity of the ferromagnetic material. From Eqs. (2) and (3), we can see that P_e is dominant in high frequency compared to the P_h as it is proportional to the frequency squared, while the P_h proportional to the frequency. So the increase of total P_{cv} can be attributed to the increase of eddy current loss which is the main component of P_{cv} at high frequency [27].

The DC-bias field dependence of the percent permeability, which is defined by the percentage of the permeability upon DC-bias field to the permeability in no DC-bias field [28], of the $Fe_{78}Si_9B_{13}$ amorphous powder cores are shown in Fig. 6. It can be seen that all the cores show superior DC-bias properties higher than 65% permeability at $H = 100$ Oe. And the cores annealed at an external transverse magnetic field of 0.5 T just change a little compared with zero field powder cores. The DC-bias property is important to our materials, because almost all powder cores are used in a DC-bias field. The DC-bias properties can be explained by magnetizing. If there have more air gaps into the powder cores, we can say it has a good DC-bias property. Air gaps can pin the domain wall in the magnetizing process, then suppress the decrease of permeability. On the other hand, the well-distributed resin between the Fe-Si-B amorphous powders can separate the powders electrically from each other, resulting in the reduction of eddy current loss in high frequency range and the stable permeability. The high percent permeability implies that the Fe-Si-B amorphous powder cores were not easily saturated under the applied fields.

4. Conclusions

Transverse magnetic field annealing is proved to be effective in improving the soft magnetic properties of amorphous powder cores. When the cores annealed at 400 °C under a transverse magnetic field of 0.5 T for 30 min, the permeability was improved from 85 to 90 compared with zero field annealing, while the core loss was decreased from 260 to 235 W/kg. Meanwhile, there is no distinguishable change on the DC bias property of the magnetic powder cores. The improvement of soft magnetic properties of ordered structures is helpful for optimal design of magnetic composite cores toward practical applications.

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