

Article

Effect of Stress on Magnetic Property of the SiO₂-Added MnZn Ferrites

Yao Ying ^{1,2,*}, Yihao Zhu ^{1,2}, Jingwu Zheng ^{1,2} , Jing Yu ^{1,2}, Liang Qiao ^{1,2} , Juan Li ^{1,2} , Naoki Wakiya ³  and Shenglei Che ^{1,2,*} 

¹ College of Materials Science and Engineering, Zhejiang University of Technology, Hangzhou 310014, China

² Research Center of Magnetic and Electronic Materials, Zhejiang University of Technology, Hangzhou 310014, China

³ Research Institute of Electronics, Shizuoka University, Shizuoka 422-8526, Japan

* Correspondence: yying@zjut.edu.cn (Y.Y.); cheshenglei@zjut.edu.cn (S.C.)

Abstract

In this work, the SiO₂-added MnZn power ferrites have been prepared by the conventional ceramic method, and the effect of stress on initial permeability and power loss has been investigated. With increasing SiO₂ content, initial permeability firstly increases and then decreases, whereas power loss firstly decreases and then increases. The sample with 50 ppm SiO₂ additive exhibits optimal magnetic performance, including the highest initial permeability and the lowest power loss. This optimal sample also exhibits the wide-temperature characteristics of power loss. Initial permeability decreases and power loss increases under the applied stress. The sample with 75 ppm SiO₂ additive exhibits the best stress insensitivity of initial permeability and power loss. Through the loss separation method, it is revealed that magnetic hysteresis loss is more sensitive whereas eddy current loss remains almost unchanged with stress. An appropriate addition of SiO₂ reduces the stress sensitivity of the initial permeability and power loss of MnZn ferrites.

Keywords: MnZn ferrite; power loss; initial permeability; stress sensitivity

1. Introduction

MnZn ferrites are the most manufactured and widely used soft ferrites and serve as the core materials in electronic components such as transformers, inductors, convertors, inverters and noise filters [1–6]. In the past decade, the increasing applications in electric vehicles, 5G communication and outdoor solar have put forward new requirements, such as maintaining low power loss (P_{cv}) over a wide temperature range, for MnZn ferrites [7]. In general, P_{cv} comprises magnetic hysteresis loss (P_{hv}), eddy current loss (P_{ev}) and residual loss (P_{rv}) based on different generated mechanisms. It is essential to reduce these composed losses (P_{hv} , P_{ev} and P_{rv}) to develop low P_{cv} MnZn ferrites. MnZn ferrites are subjected to various external stresses in different application scenarios including the packaging, the winding of copper wire, the drying of surface coatings, etc. [8,9]. In addition, internal stress is also caused by pores, impurities and crystalline inhomogeneity at the micro-scale. The magnetic property of MnZn ferrites is sensitive to stress [10–15]. Previous works investigated the influence of stress on initial permeability (μ_i) and revealed that μ_i generally decreased and the temperature of the secondary permeability peak shifted under stress [10–12]. Other works also demonstrated that the applied stress generally degrades magnetic performance [13–15]. However, comprehensive investigation into the



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effect of stress on P_{cv} of MnZn ferrites remains limited. In particular, investigations into the effect of stress on three individual losses (P_{hv} , P_{ev} and P_{rv}) are few. Stress sensitivity can be mitigated through improving the microstructure by optimizing the sintering process, and changing the magnetostriction coefficient (λ_s) by adjusting the main composition and additives [14,16,17]. The use of additives during the sintering process is an effective strategy for tailoring the magnetic property of MnZn ferrites [18–20]. SiO_2 is a widely used additive to improve the magnetic performance by improving the microstructure and forming a high-resistivity grain boundary [21]. It should be noted that although many works have optimized SiO_2 content to enhance the soft magnetic performance of MnZn ferrites, the influence of SiO_2 additive on the stress sensitivity of P_{cv} is still unclear. In particular, few works have quantitatively investigated the role of SiO_2 additive in suppressing the stress sensitivity of P_{hv} and P_{ev} .

In this work, the MnZn ferrites with the wide-temperature characteristic have been prepared by adding the SiO_2 additive. The effects of stress on μ_i and P_{cv} were systematically evaluated. Loss separation was performed to elucidate the influence of stress on individual losses (P_{hv} and P_{ev}). It has been found that stress degrades both μ_i and P_{cv} . According to the result of loss separation, the increase in P_{cv} under stress is mainly ascribed to the increase in P_{hv} . It is also found that the optimal magnetic performance appears in the sample with 50 ppm SiO_2 whereas the best stress insensitivity appears in the sample with 75 ppm SiO_2 . The choice of samples with 50 or 75 ppm SiO_2 depends on application requirements.

2. Experimental

$Mn_{0.7}Zn_{0.2}Fe_{2.1}O_4$ ferrites with SiO_2 additive were synthesized via a conventional solid state reaction method. First, raw materials (Fe_2O_3 , Mn_3O_4 , and ZnO) were weighed according to the stoichiometric ratio, mixed with deionized water, and ball milled for 16 h. The mixture was then dried and pre-sintered at 850 °C for 3 h. Subsequently, additives including SiO_2 , $CaCO_3$ and Co_3O_4 were added to the calcined powder and subjected to a secondary ball-milling process. The samples with 25, 50, 75, 100 and 125 ppm SiO_2 were named as Si25, Si50, Si75, Si100 and Si125, respectively. The resulting powder was dried, granulated with an 8 wt% polyvinyl alcohol (PVA) solution, and pressed into toroidal green bodies under a specific stress. Finally, samples were sintered at 1270 °C and cooled under equilibrium oxygen partial stress.

The crystal structure of sintered samples was analyzed by using X-ray diffraction (XRD, Rigaku Ultima with K_α radiation of copper, Malvern Panalytical, Almelo, The Netherlands). The fracture surface morphology was examined by using scanning electron microscopy (SEM, Hitachi SU1510, Tokyo, Japan), and the average grain size (D) was estimated via the linear intercept method. The bulk density was measured by the Archimedes drainage method. Initial permeability was measured with an LCR meter (Agilent E4991A, Santa Clara, CA, USA). Power loss under varying frequency, stress, and temperature were measured by using a B-H analyzer (IWATSU SY-8218, Tokyo, Japan). The complex impedance spectra of samples under various compressive stress were measured by using the electrochemical workstation (Zennium, ZAHNER, Kronach, Germany). During the stress measurement, the stress gauge was employed to apply a uniaxial compressive stress to the sample. The direction of loading was perpendicular to the top and bottom surfaces of the sample.

3. Results and Discussion

Figure 1 shows the XRD patterns of MnZn ferrites with different SiO_2 content. The XRD spectra of all samples were compared with the standard card (PDF#74-2402). The diffraction peaks of all samples are in accord with the characteristic peaks of the standard

card and there are no other heterogeneous peaks, indicating that all samples present single spinel phases with no heterogeneous phases.

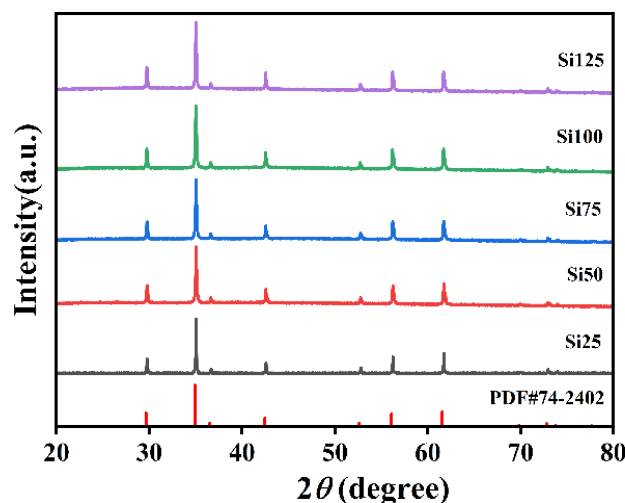


Figure 1. XRD patterns of MnZn ferrites with different SiO₂ content.

Figure 2a–e show SEM images of MnZn ferrites with different SiO₂ content, and Figure 2f–i present the grain size distribution of samples Si25, Si50, Si75 and Si100, respectively. The average grain sizes of samples have been estimated and presented in Table 1. The increase in SiO₂ content slightly promotes grain growth. This growth is attributed to the formation of a low-eutectic liquid phase at grain boundaries during the sintering process, resulting from the reaction between SiO₂ and CaO [22,23]. This liquid phase acts as a rapid diffusion channel, facilitating atomic and ionic migration and thereby accelerating grain boundary movement. However, when the SiO₂ content becomes excessive (such as 125 ppm), grains become too large to estimate the average grain size, which reference the occurrence of abnormal grain growth (Figure 2e). The formed excessive liquid phase displaces other additives segregated at grain boundaries, weakening their pinning effect on grain boundary motion [24,25]. Furthermore, the unevenly distributed liquid phase induces the different grain boundary migration rate in different regions, which leads to abnormal grain growth.

Table 1. Average grain size and density of MnZn ferrite samples with different SiO₂ content.

S samples	SiO ₂ Content (ppm)	Average Grain Size (μm)	Density (g/cm ³)
Si25	25	7.1 ± 2.4	4.92
Si50	50	7.3 ± 2.0	4.92
Si75	75	7.5 ± 2.4	4.93
Si100	100	7.6 ± 2.9	4.93
Si125	125	/	4.93

Figure 3 illustrates temperature dependence of initial permeability [$\mu_i(T)$] of MnZn ferrites with different SiO₂ content. At 25 °C, μ_i initially increases and then decreases with increasing SiO₂ content. μ_i , as a key parameter characterizing the macroscopic response of a soft magnetic material to weak-field magnetization, is microscopically determined by the combined contributions of domain wall displacement and domain rotation [26]. The observed trend can be attributed to competing factors. First, the increased SiO₂ content promotes grain growth. Larger grains accommodate more magnetic domain, reduce the impeding effect of grain boundaries on domain wall motion, and consequently enhances the

domain wall displacement contribution to magnetization, which is beneficial to improve μ_i . When the SiO_2 content exceeds an optimal value, it induces non-uniform grain distribution and lattice distortion. This leads to the decrease in μ_i .

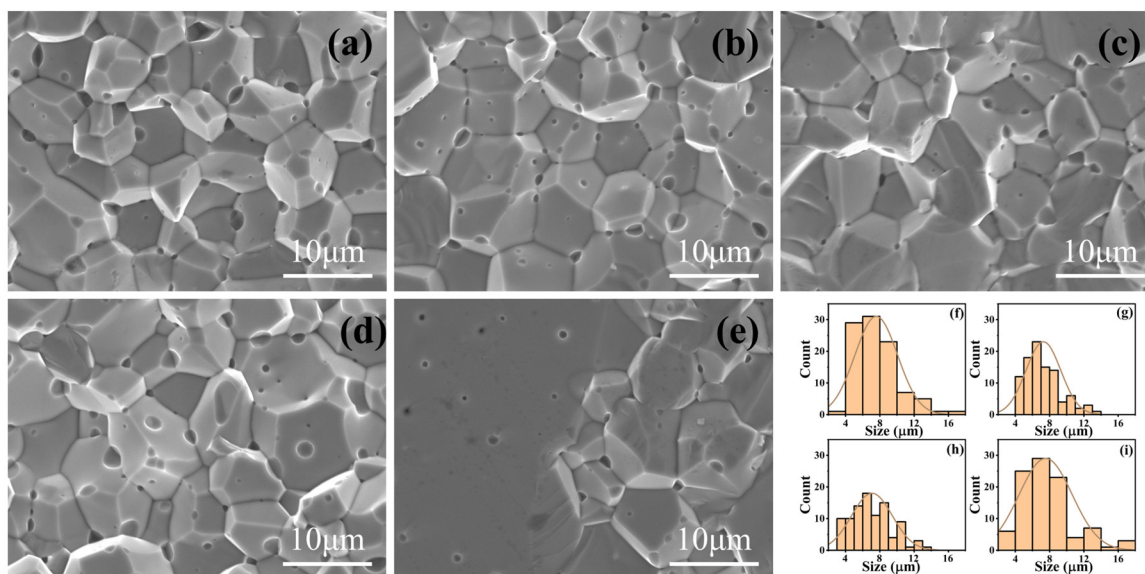


Figure 2. SEM images of MnZn ferrites with different SiO_2 content: (a) Si25, (b) Si50, (c) Si75, (d) Si100 and (e) Si125. Figures (f–i) present the grain size distribution of samples Si25, Si50, Si75 and Si100.

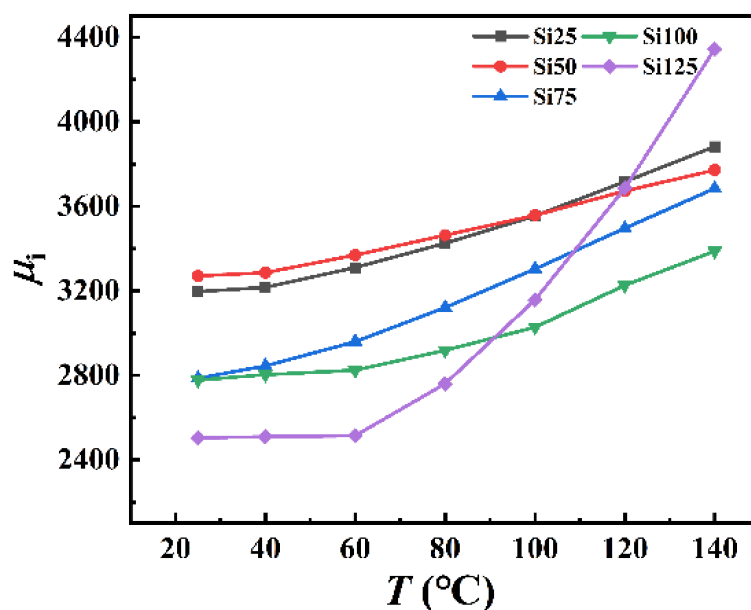


Figure 3. Temperature dependence of initial permeability of MnZn ferrites with different SiO_2 content.

Figure 4 presents the temperature dependence of power loss [$P_{cv}(T)$] at 100 kHz/200 mT and 300 kHz/100 mT for MnZn ferrites with different SiO_2 content. Sample Si25 exhibits that the P_{cv} remains low in a wide temperature range from 25 to 140 °C, reflecting the excellent temperature stability. When the SiO_2 content increases from 25 to 50 ppm, the P_{cv} of sample Si50 decreases in the whole temperature range compared to sample Si25. When the SiO_2 content further increases, the P_{cv} oppositely increases. This optimal sample Si50 shows that the P_{cv} is 329.4 kW/m³ (100 kHz/200 mT) at 25 °C and remains less than 432.5 kW/m³ in the whole temperature range. The excellent temperature stability is primarily attributed to the introduction of Co^{2+} ions [27,28]. The magnetocrystalline

anisotropy constant (K_1) of MnZn ferrite is negative, whereas that of Co^{2+} is positive. Therefore, an appropriate addition of Co^{2+} can compensate for the negative K_1 of the matrix, creating two temperature points where K_1 is zero [7,27,28], as shown in Figure 4c. Between these compensation points, the absolute value of K_1 remains small, which results in low P_{hv} and subsequently low P_{cv} in the wide temperature range, characteristics of temperature stability. As seen from the $P_{\text{cv}}(T)$ curves (Figure 4c) two $K_1 = 0$ points seem to appear at around 25 and 100 °C. This is in agreement with the previous result in [7].

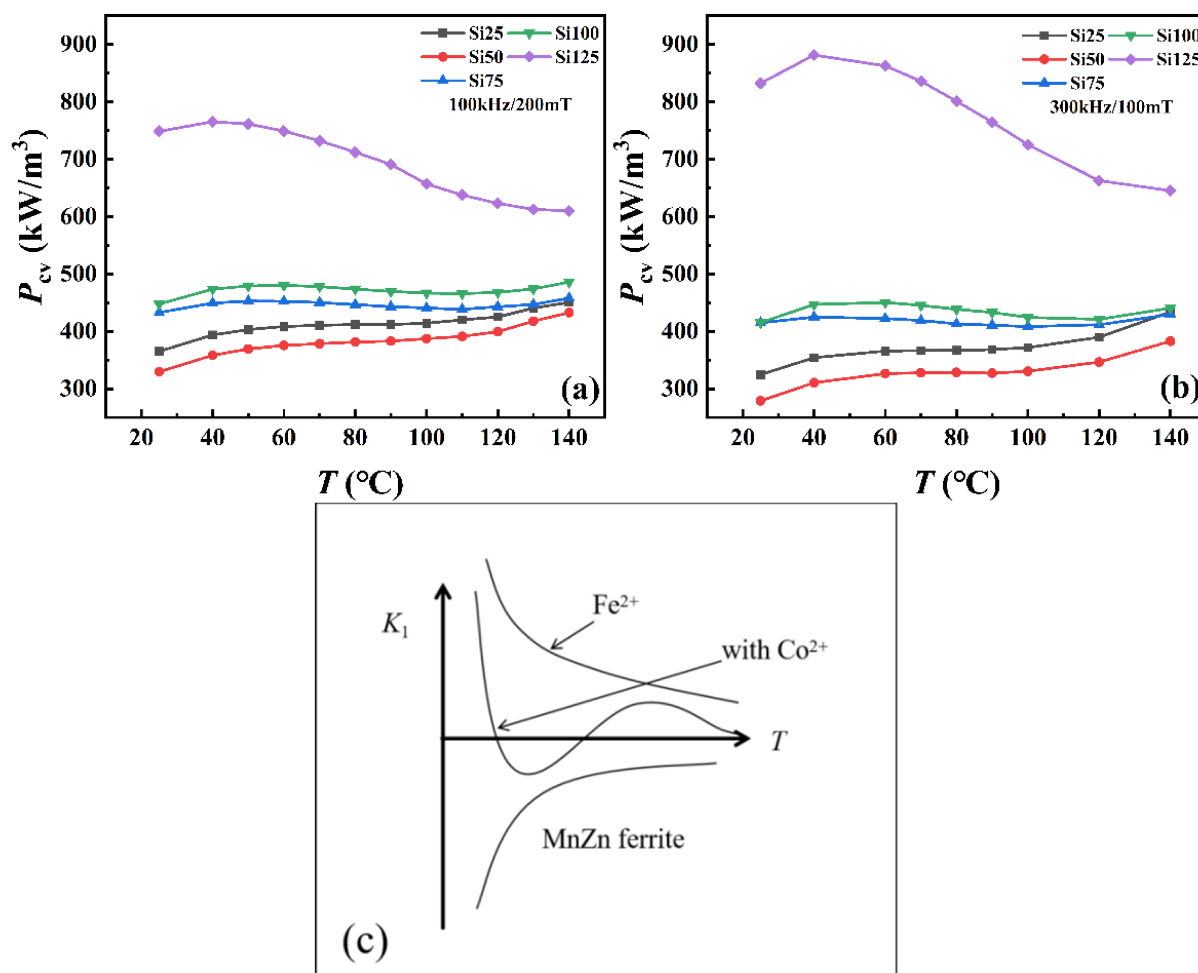


Figure 4. Temperature dependence of power loss for MnZn ferrites with different SiO_2 content under the measurement conditions of (a) 100 kHz/200 mT and (b) 300 kHz/100 mT. (c) Schematic drawing of K_1 as a function of temperature for MnZn ferrites with the contribution of Co^{2+} .

P_{cv} comprises P_{hv} , P_{ev} and P_{rv} . In the low and medium frequency condition, P_{rv} is negligible. To further clarify the effect of the SiO_2 addition on P_{cv} , P_{cv} at 100 kHz/200 mT and 300 kHz/100 mT, 25 °C was separated into P_{hv} and P_{ev} , and expressed as:

$$P_{\text{cv}} = P_{\text{hv}} + P_{\text{ev}} = K_{\text{H}}B_{\text{m}}^3f + \frac{K_{\text{E}}B_{\text{m}}^2f^2}{\rho} \quad (1)$$

where K_{H} and K_{E} are parameters, f is frequency, B_{m} is the maximum magnetic flux density, and ρ is resistivity. In order to separate these losses, P_{cv} s were measured at the same B_{m} under different frequencies. Then the P_{cv} data were linearly fitted by Equation (1) divided by f . P_{hv} was calculated from the intercept, and P_{ev} was calculated from the remained loss, respectively. The loss separation results are shown in Figure 5. At 100 kHz, the contribution of P_{hv} to P_{cv} exceeds that of P_{ev} . When f increases to 300 kHz, P_{ev}

becomes more predominant. As shown in Equation (1), P_{hv} is proportional to f , whereas P_{ev} is proportional to f^2 . Therefore, P_{ev} increases faster with increasing f than P_{hv} , and subsequently becomes more predominant at 300 kHz. Figure 6 shows the variation in P_{hv} and μ_i with the SiO_2 content. The variation tendencies of P_{hv} and μ_i are opposite. With increasing the SiO_2 content, P_{hv} first decreases and then increases whereas μ_i first increases and then decreases, which is in agreement with the previous work [29]. The minimum P_{hv} and the maximum μ_i appears in the sample with 50 ppm SiO_2 . This is due to the fact that the liquid phase was formed by the SiO_2 addition during the sintering process. The formed liquid phase promotes grain growth and densification. The larger grains facilitate magnetic domain wall displacement and thereby increase μ_i and reduce P_{hv} . However, with excessive SiO_2 content, the abnormal grain growth forms abnormal large grains with many pores in the grains. Pores are non-magnetic defects. According to magnetic domain theory, when a domain wall encounters a pore, system energy is minimal due to the low domain wall energy at the pore, so the domain wall is “pinned” at the pore. To drive the domain wall motion, it requires additional energy to overcome the pinning force. Consequently, the presence of intragranular pores reduces μ_i and increases P_{hv} .

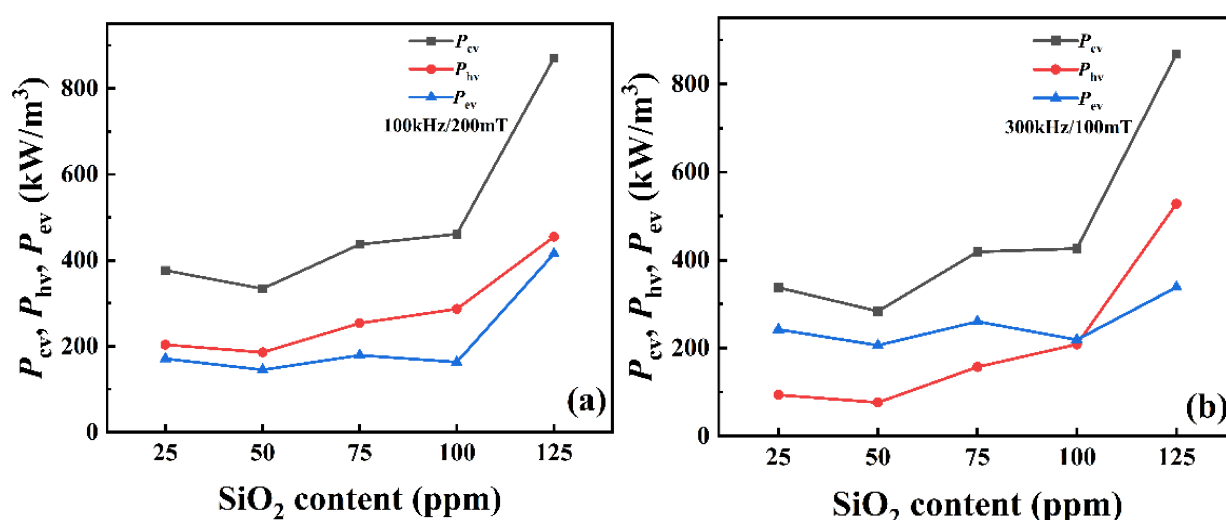


Figure 5. Loss separation results of MnZn ferrites with different SiO_2 content at 25 °C: (a) 100 kHz/200 mT and (b) 300 kHz/100 mT.

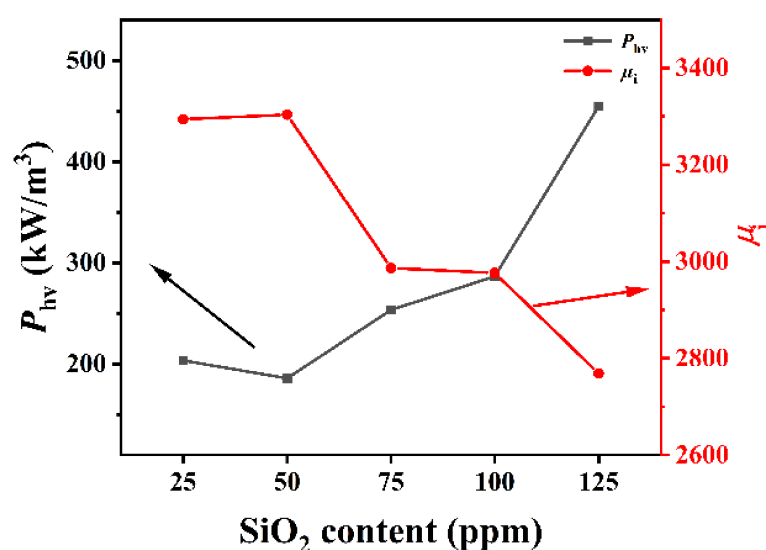


Figure 6. Variation in P_{hv} and μ_i with the SiO_2 content.

We have further investigated the effect of compressive stress on the magnetic property of MnZn ferrites. We select a compressive stress range of 0–0.18 MPa. The selection of this range is informed by the stress intervals reported in the relevant literature for ferrites exhibiting low stress sensitivity. Under these conditions, the initial magnetic permeability and power loss demonstrate a significant deterioration trend. Furthermore, this stress range corresponds to the typical magnitude of mechanical stresses generated by core winding and clamping in practical applications, making it highly representative [12]. Figure 7 shows the variation in μ_i with the applied stress at 25 °C. For all samples, μ_i decreases with increasing stress. When the applied stress reaches 0.18 MPa, the μ_i of the Si25 sample decreases from 3263 to 2945. It is noted that the μ_i of the Si75 sample shows the most insensitivity with the applied stress. Theoretically, the μ_i is inversely proportional to the product of magnetostriction coefficient (λ_s) and stress (σ) and also related to magnetocrystalline anisotropy constant [30,31]. The decrease in μ_i may be related to an increase in stress anisotropy. In this series of samples, The $K_1 = 0$ point is at 25 °C. So, μ_i becomes more sensitive to stress due to the fact that magnetic moments are prone to aligning along the direction of minimum stress energy and thereby increasing stress anisotropy [14]. This enhanced anisotropy impedes domain wall displacement and domain rotation, and as a result μ_i decreases. Coercivity (H_c) of samples Si50 and Si75 under different stress is also shown in Table 2. H_c of sample Si50 is lower than that of Si75, which is in accord with the low P_{hv} in the sample Si50. For both samples Si50 and Si75, H_c increases with the applied stress. The variation in H_c under stress in sample Si75 is less than that in sample Si50, which may result in the lower stress sensitivity of power loss in sample Si75.

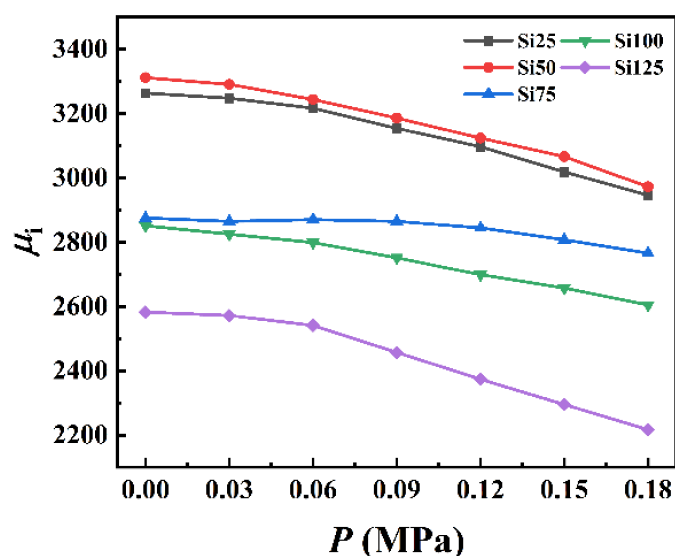


Figure 7. Variation in initial permeability with stress for MnZn ferrites with different SiO₂ content.

Table 2. Coercivity of samples Si50 and Si75 under different stress.

Stress (MPa)		0.03	0.06	0.09	0.12	0.15	0.18
H_c (A/m)	Si50	5.73	6.12	6.55	6.87	7.41	7.92
	Si75	7.48	7.52	7.60	7.78	8.01	8.35

Figure 8 presents the variation in P_{cv} with applied stress for MnZn ferrites with different SiO₂ content at 25 °C. It is shown that P_{cv} increases with stress for all samples. Table 3 shows the variation in relative change in P_{cv} under 0.18 MPa for all samples. It is noted that the P_{cv} of the Si75 sample shows the most insensitivity with the applied stress, which is in accord with the μ_i behavior. Thus, an appropriate addition of SiO₂ (75 ppm) can

reduce the stress sensitivity of magnetic property in MnZn ferrites. The P_{cv} at 0.18 MPa was further separated to P_{hv} and P_{ev} , shown in Figure 9. It reveals that P_{hv} remains dominant in P_{cv} under stress. As stress increases, both P_{hv} and P_{ev} increase for all samples. Compressive stress generates localized anisotropic stress, which hinders domain wall displacement. This results in the increase in P_{hv} . The increase in P_{cv} under compressive stress is mostly ascribed to the increase in P_{hv} . As shown in Equation (1), P_{ev} is inversely proportional to resistivity. To further illustrate the mechanism of stress on P_{ev} , complex impedance spectra of sample Si75 under different stresses were measured. As shown in Figure 10, the complex impedance curves of all samples show relatively semicircles. According to the viewpoint of electricity, resistance in polycrystalline ferrite can be described with the equivalent circuit model [7,32]. There are two intercepts of the real part of impedance (Z') appearing in the complex impedance curve. The high intercept value, corresponding to the dc current response, is equal to the sum of grain resistance (R_g) and grain boundary resistance (R_{gb}). The low intercept value is equal to R_g , corresponding to that the grain boundaries are short-circuited by the high-frequency ac current. As the stress increases, the high intercept value decreases monotonically, which indicates that stress reduces the material's resistance. In order to clarify the effect of stress on R_g and R_{gb} , the equivalent circuit model was used to fit the complex impedance (inset of Figure 10). Figure 11 illustrates R_{gb} and R_g for the Si75 sample under various compressive stresses. It is shown that both R_{gb} and R_g (also meaning grain boundary resistivity and grain resistivity) decrease with increasing the applied stress. The stress acting on grains will reduce the distance between Fe^{3+} and Fe^{2+} ions, which is in favor of the electron hopping between Fe^{3+} and Fe^{2+} ions and leads to the decrease in grain resistivity. When the applied compressive stress increases, grain boundaries become thinner. This enhances the electron quantum tunneling effect between adjacent grains and consequently reduces grain boundary resistivity, which is in agreement with the result in [33]. The decrease in grain boundary resistivity and grain resistivity accounts for the increase in P_{ev} under the compressive stress.

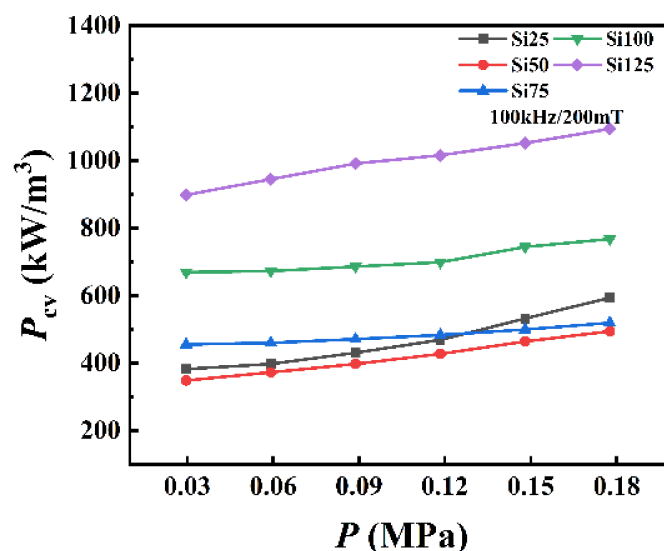


Figure 8. Variation in P_{cv} with stress for MnZn ferrites with different SiO_2 content.

Table 3. Relative change (%) in P_{cv} under the stress of 0.18 MPa for all samples.

Relative Change (%)	Si25	Si50	Si75	Si100	Si125
P_{cv}	55.40	41.84	14.03	14.74	21.80

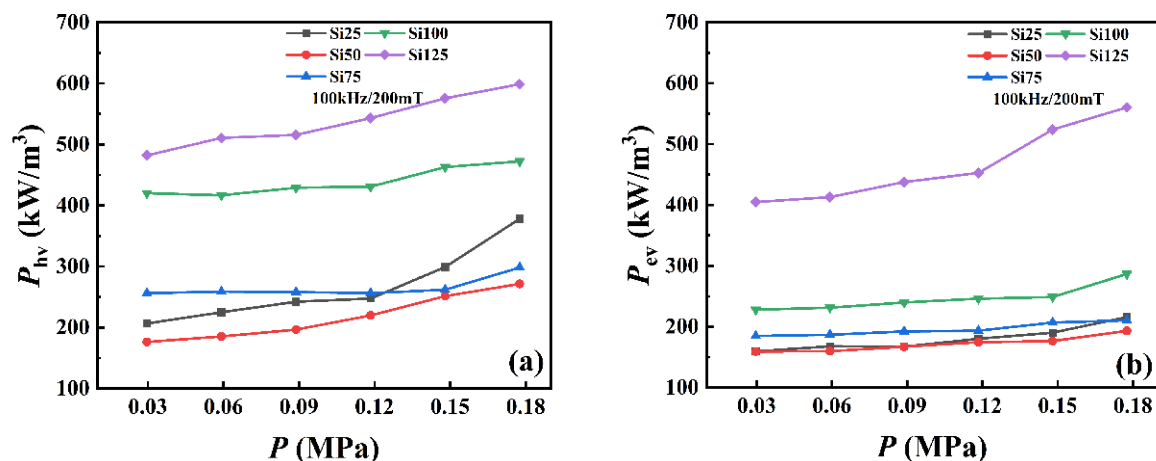


Figure 9. Variation in (a) P_{hv} and (b) P_{ev} with stress for MnZn ferrites with different SiO_2 content.

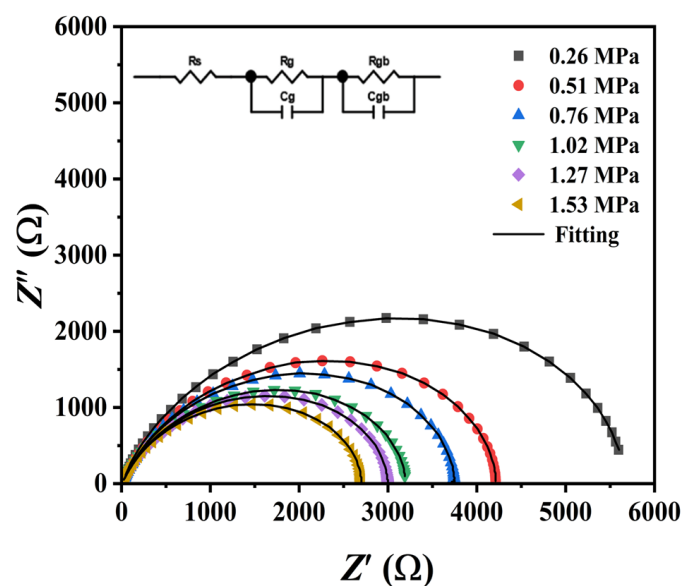


Figure 10. Complex impedance spectra for the Si75 sample under different compressive stress.

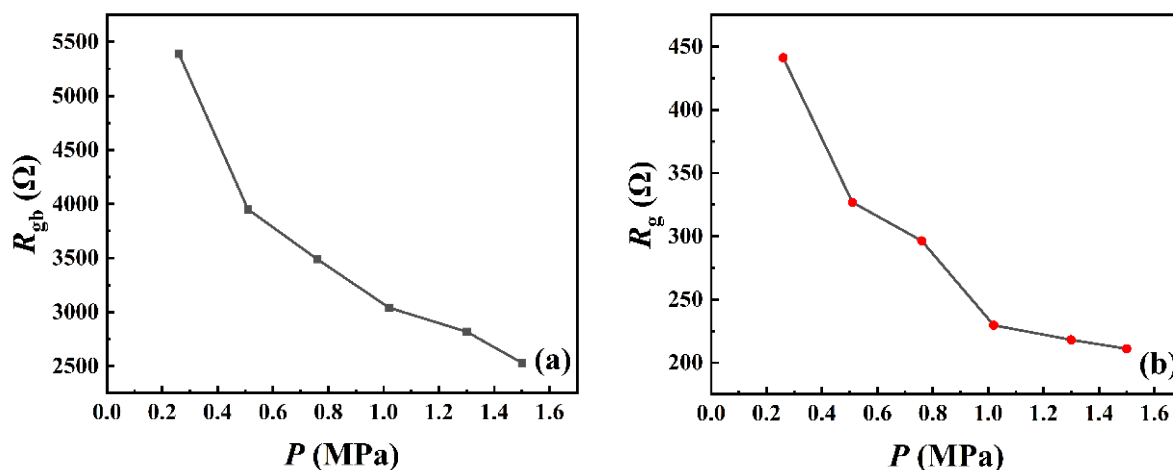


Figure 11. (a) R_{gb} and (b) R_g of the Si75 sample under various compressive stress.

4. Conclusions

In this work, a wide-temperature low-loss MnZn ferrite was prepared by optimizing the SiO_2 content. The sample with the 50 ppm SiO_2 additive shows optimal power loss

performance with P_{cv} s at 100 kHz/200 mT and 300 kHz/100 mT, with 25 °C being 329.42 and 278.97 kW/m³ and maintaining less than 432.54 and 382.74 kW/m³ in the temperature range from 25 to 140 °C, respectively. The effect of compressive stress on μ_i and P_{cv} was investigated. μ_i decreases and P_{cv} increases under compressive stress. An appropriate addition of SiO₂ reduces the stress sensitivity of μ_i and P_{cv} . It is revealed that P_{hv} is more sensitive, whereas P_{ev} remains almost unchanged with stress. The increase in P_{cv} under stress mainly originates from the increase in P_{hv} . The sample with the 50 ppm SiO₂ additive shows the optimal power loss performance, whereas the sample with the 75 ppm SiO₂ additive exhibits the more insensitivity of stress. The choice of samples Si50 and Si75 depends on application requirements. Sample Si75 may be more suitable in the application scenarios of external stress.

Due to the limitations of the experiment condition, the resistivity under stress could not be directly measured. In this work, the change in R_{gb} and R_g under stress was obtained by fitting the complex impedance with the equivalent circuit model, which well explains the variation in P_{ev} under stress. The magnetostriction coefficient is also not available due to the limitation of the experiment conditions. The magnetostriction coefficient is important for the further quantitative analysis of stress effect on magnetic properties.

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