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ARTICLE

Improvement of the Piezomagnetic Measurement and Characterization Method of Fe-based Amorphous Alloy Ribbons and Their Piezomagnetic Properties

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Abstract: A new improved test system was developed based on the non-contact inductance method, which can be used to characterize the piezomagnetic properties of Fe-based amorphous alloy ribbons properly. The studies show that when the test frequency is 1 kHz, the voltage is 0.3 V and the compressive stress $\sigma \leq$ 0.1 MPa, the amorphous magnetic material of $Fe_{73.5}Cu_1Nb_3Si_{13.5}B_9$ has excellent piezomagnetic properties, and its ribbons of different specifications exhibit perfect stability and repeatability after repeated experiments. The ribbons are extremely sensitive to tiny stress, and they show a rapidly rising trend of the inductance once the initial pressure is applied on the ribbons. Moreover, the further amplifying experiment indicates that the sensitivity range is 0~1.5 kPa. In addition, the size of $Fe_{73.5}Cu_1Nb_3Si_{13.5}B_9$ ribbons influence significantly the piezomagnetic property, and the initial inductance and its corresponding $S_{\text{I-Max}}$ value of the ribbons increase substantially along with the sample size increasing; with the same width, the piezomagnetic property is superior to others while the thickness is 33~36 μm, and $S_{\text{I-Max}}$ is 19.8%. With the same thickness, the ribbons with a width of 20 mm show an excellent piezomagnetic property and the $S_{\text{I-Max}}$ has reached 22.02%.

Key words: amorphous alloy; piezomagnetic effect; force-sensing property; sensing element

Fe-based amorphous alloy is a new material made by a special metallurgical technique, named rapid quenching method, which has been widely used in electricity and electronics field due to its excellent magnetic properties[1-7]. In addition, due to the outstanding piezomagnetic performance, force-sensitive sensors based on it have attracted increasing attention in recent years[8-15].

The piezomagnetic effect describes the phenomenon which characterizes the strength of the variation of permeability μ as stress force σ is imposed on the surface of amorphous ribbon [16]. Generally, the usual experimental way to characterize the piezomagnetic effect is to measure the saturation magnetostriction coefficient λ_s [15]. However, for the limitations of measurement method, the results may be somewhat unreliable; what's worse it can't directly reflect the corresponding change of magnetic properties and stress.

We have conducted researches on this direction since 2003. Based on the testing method of force-sensitive film, we tried to improve the testing methods gradually. With the original testing method [16-21], the samples are cut into ribbons and the both besides of a ribbon is polished to rough surfaces with 5 mm; then wires were welded on the polished samples and the impedance values between two wires were measured to characterize the piezomagnetic property (Fig. 1). This method can be used to characterize the piezomagnetic property of Fe-based amorphous alloy ribbon, but the measuring accuracy is limited. Additionally, the welding process is difficult to control and the encapsulate problem is hard to solve. In our previous works[20,21], the piezomagnetic effect of Fe-based amorphous alloy ribbon was studied using the non-contact

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inductive method by applying continuous stress on the surface of the ribbon and testing the variations of the nearby closed loop inductance (Fig. 2). This method can effectively solve the encapsulation and low accuracy problems when the Fe-based amorphous alloy ribbon is applied as a sensor element. However, there are still some deficiencies in the system, such as slow responsibility and sensitivity to the surrounding fluctuations.

In this paper, a new test system was developed based on the non-contact inductive method which was proposed by Du Kang etc. We chose the closed loop inductance value changes to characterize the piezomagnetic property of Fe-based amorphous alloys ribbon; while the ribbon would be directly tested as an excitation coil magnetic core in our device, and the piezomagnetic properties were characterized by measuring the whole closed loop inductance value, including the excitation coil magnetic core produced by ribbon and the measuring device. Through the improvement of this testing system, the advantages of non-contact inductive methods were retained; meanwhile, the anti-interference ability was obviously enhanced. The closed loop was significantly influenced by the internal magnetic core, and the results can more intuitively and accurately reflect the piezomagnetic property of the ribbons.

1 Testing Methods and Analysis

1.1 Experimental samples

$Fe_{73.5}Cu_1Nb_3Si_{13.5}B_9$ amorphous alloy ribbons were prepared by a rapid quenching method (see Fig. 3), The ribbon with good surface condition remained its state of the original quenching, and the length of the ribbon is 100 mm. The phase composition of the ribbon was investigated by X-ray diffraction (XRD) (D8 Advance, Bruker-axe, Germany) using Cu Kα radiation (as shown in Fig. 4). According to the results, the diffraction angle is 10°~90°, and there are no obvious crystallization peaks during the entire process of diffraction. In the vicinity of $2\theta = 45°$, a diffuse peak appears in the XRD patterns which indicates that the $Fe_{73.5}Cu_1Nb_3Si_{13.5}B_9$ amorphous alloy ribbon in quenching condition is a single amorphous structure.

1.2 Piezomagnetic property testing and characterization method

Fig. 5 is the schematic diagram of our improved testing

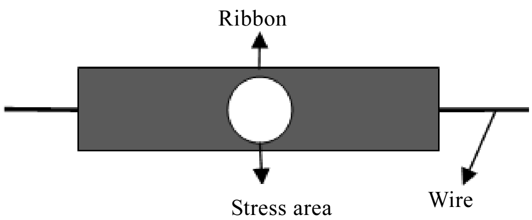

Fig. 1 Schematic diagram of the impedance testing method

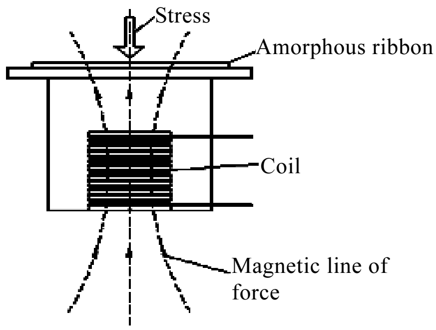

Fig. 2 Schematic diagram of the non-contact inductive method

device which is mainly made up of two sets of excitation coils connected in series. The magnetic field lines are assembled along internal of the testing ribbon, so as to improve the reliability and stability of the testing results. Fig. 6 shows the equivalent circuit of the testing device.

In order to meet the different sizes of testing, its internal plate glass and the load pressure head were designed as detachable units. In the traditional testing method, the surface of the ribbon usually covered with a non-magnetic plate to ensure the accuracy of the test results does not be affected by the flatness. However, the non-magnetic plate may make it difficult to determine the sensitive range: as a matter of fact, the ribbon is very sensitive to tiny stress, while the plate glass is equivalent to a certain preloading force applied on the ribbon, and this preloading force is likely to affect even beyond the sensitive range. The design of the activity unit exactly solves this problem. Before the experiment, the device is fixed on the pressure testing machine, and the machine is zero set. Then, the plate glass, loading pressure head and the testing ribbon are put on the position shown in Fig. 5 and the ribbon is adjusted again to ensure that it is in the center. The pressure tester is started to adjust the distance between the glass plate and the inner wall of the device. The testing machine is set to stop when the stress is greater than zero. At this point, the position of the plate glass and the inner wall of the device are just in contact with each other. The testing ribbon is in a flat state as limited by the upper and lower space but no over-force exists. Reset the machine to zero again, and then the formal

Fig. 3 Picture of Fe-based amorphous ribbon products

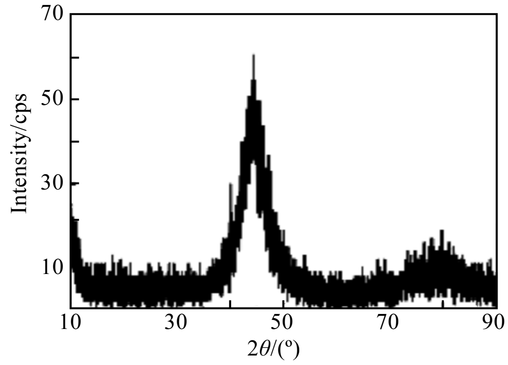

Fig.4 XRD pattern of as-quenched $Fe_{73.5}Cu_1Nb_3Si_{13.5}B_9$ amorphous alloy ribbon

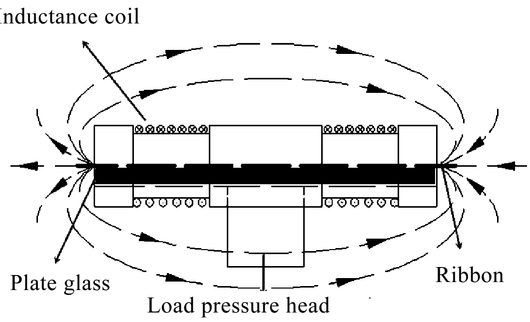

Fig. 5 Schematic diagram of the testing device

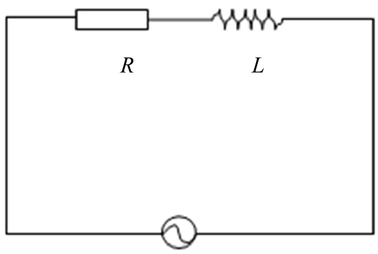

Fig. 6 Equivalent circuit of the testing device

testing work can be carried out.

The loading test was carried out by LYYL-500N pressure testing machine; at the same time, inductance L_s was collected by TH-2816B precision digital electric bridge. We operated our experiments under the testing conditions as follows: testing frequency f = 1 kHz, testing voltage = 0.3 V; the pressure testing machine loads in the range of 0~0.1 MPa and the loading speed is 0.1 mm/min.

The piezomagnetic effect was characterized using L_s-σ curves. The piezomagnetic effect value S_I is defined as:

$$S_1 = \frac{\Delta L_s}{L_s} = \frac{L_s(\sigma) - L_s(\sigma_0)}{L_s(\sigma_0)} \times 100\% \quad (1)$$

where L_s is the coil inductance value, $L_s(\sigma)$ is the coil inductance when the compressive stress is equal to σ, and the $L_s(\sigma_0)$ is the inductance value with no compressive stress. $Fe_{73.5}Cu_1Nb_3Si_{13.5}B_9$ amorphous alloy ribbon with good surface condition was prepared in our experiment. In addition, the ribbons were tested with different thickness and widths to explore the piezomagnetic effect.

2 Piezomagnetic Property of $Fe_{73.5}Cu_1Nb_3Si_{13.5}B_9$ Amorphous Ribbons

Fig.7 shows the curves of piezomagnetic property of Fe-based amorphous ribbon with different testing sequences. (Results of different testing sequence are marked by black squares, red circles, and blue triangles) The size of the testing ribbon is 8 mm×100 mm, and the thickness is 18~21 μm. During the first 100 s, the inductance increases gradually with compressive stress increasing, while the variation of the stress applied on the ribbon is very small, and there is a significant inflection point at about 100 s, after which, the stress becomes somewhat lager under the time evolution, until the stress is gradually close to 0.1 MPa. Correspondingly, the inductance L_s is significantly increased to 50.282 μH, i.e. an increment of 0.772 μH. Fortunately, the stability and repeatability perform pretty well after several repetitions. We have made a comparison with the results produced by the non-contact inductive testing device. The two kinds of devices present a similar variation trend and both of them exhibit a good piezomagnetic property. However, the results show that the device in this paper is more accurate and sensitive to the piezomagnetic variation.

Fig.8 shows the curves of piezomagnetic property of Fe-based amorphous ribbon. We can observe the ribbon measured by the improved testing device presents an excellent sensitivity of piezomagnetic property over the stress, especially in the week regime. The S_I value shows a rapid rise once loaded the stress, with the stress kept in the range of 0~0.33 kPa. It indicates that the ribbon is very sensitive to tiny stress, some small changes would cause a significant variation of the inductance, and this small range which is able to perceive small changes is called the stress sensitivity range.

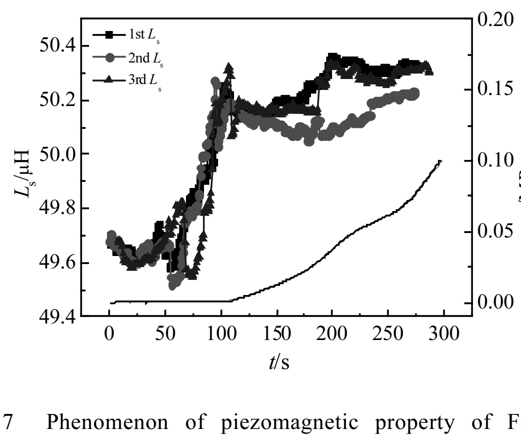

Fig. 7 Phenomenon of piezomagnetic property of Fe-based amorphous ribbon varying over time

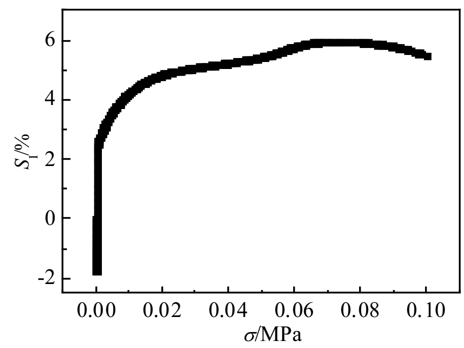

Fig.8 Curve of piezomagnetic property of Fe-based amorphous ribbon

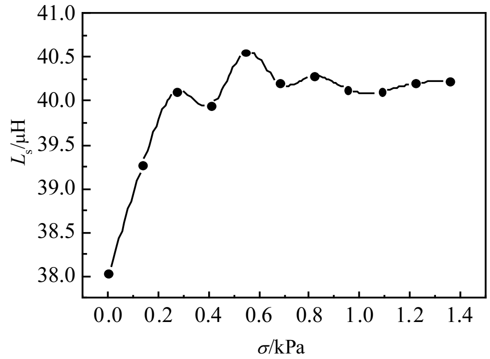

Fig.9 Curve of the inductance vs the stress of $Fe_{73.5}Cu_1Nb_3Si_{13.5}B_9$ ribbon in sensitive range

After the sensitive range, the inductance value of the ribbon will still have the corresponding change with the stress increasing, but the variation amplitude is much less than that of the initial stage. Throughout the whole stress loading process, the absolute $S_{I\text{-}Max}$ is 7.8%.

We have made a further test in order to take a deep exploration about the sensitive range and the piezomagnetic effect of the ribbons (Fig.9) with an increase of the excitation coil turns. At the same time, to reduce the influences attracted by loading velocity and the hysteresis effect, a static measurement method was taken by adding weight manually. All mass used in this experiment is 10 g, and increases to 100 g one by one. The initial inductance of the ribbon is 38.035 μH, and at the beginning of the loading process, the inductance shows a significant increasing, which gradually increases to the maximum of 40.558 μH. Then with a further increase of the stress the inductance becomes stable at a value around 40.2 μH. Combined with the continuous measurement data, it would be confirmed that the sensitive range is 0~0.33 kPa.

Because of the electromagnetic interactions, when the device was pumped into an AC signal with a certain frequency, the excitation coil and its internal amorphous ribbon will produce a closed magnetic circuit, and the inductance value of the coil will change according to the increase of stress. Based on the relevant knowledge of ferromagnetics, piezomagnetic effect can be qualitatively explained by the skin effect[10]. The stress applied on the amorphous alloy ribbon can lead to a corresponding strain, and the equivalent permeability within the material will change due to the magneto-elastic coupling, thereby affecting the skin depth of the material. The expression can be given as:

$$\delta = \sqrt{\rho / \pi f \mu_{\text{eff}}} \tag{2}$$

Where f is the current frequency, ρ and μ_{eff} is the resistivity and effective permeability of the material, respectively. The impedance Z of the ribbon can be expressed as:

$$Z = (k\rho l/2w)\coth(kt/2) \tag{3}$$

where l, w and t is the length, width and thickness of the ribbons, respectively.

It indicates that the impedance Z is connected with the skin depth from equations mentioned above, while the skin depth is dependent on the effective permeability of the material, and the effective permeability directly affects the inductance of the material.

According to the mechanism of piezomagnetic effect, the main factor which determines the piezomagnetic effect is the state of the internal stress of Fe-based amorphous ribbon in the loading process, where the thickness and the width of the ribbon make a big difference. In this case, it is necessary to take further experiments to study the influence induced by the thickness and width.

First, we demonstrated the difference with taking the sample width into consideration and the result is shown in Fig.10. The specification of testing samples is 8 mm×100 mm, and the thickness of samples is 18~21, 24~28, 31~33 and 33~36 μm, respectively (Fig.10a~10d). Substantially, the ribbons of each group bring out the similar piezomagnetic phenomena. It is obvious that the initial inductance $L_s(0)$ is affected by the thickness, which increases with the increase of the thickness, and the initial value of four samples is 9.76, 16.213, 38.40 and 38.40 μH, respectively. Note that, the thickness of sample c and d are relatively closer, and the testing data detected by the instrument may have a little fluctuation in the loading process; while, the inductance value measured in this experiment is small and the interference is amplified, the initial inductance value has no obvious change. With the increase of the thickness, the inductance range L_s increases obviously, and the range of the closed loop inductance of four kinds of ribbons is 0.77, 2.41, 2.54 and 7.80 μH, respectively.

To make a direct observation of the stress sensitivity on the ribbons, we further tested piezomagnetic property of $Fe_{73.5}Cu_1Nb_3Si_{13.5}B_9$ ribbon with different thickness, which is exhibited in Fig. 11. All sets of ribbons present a similar

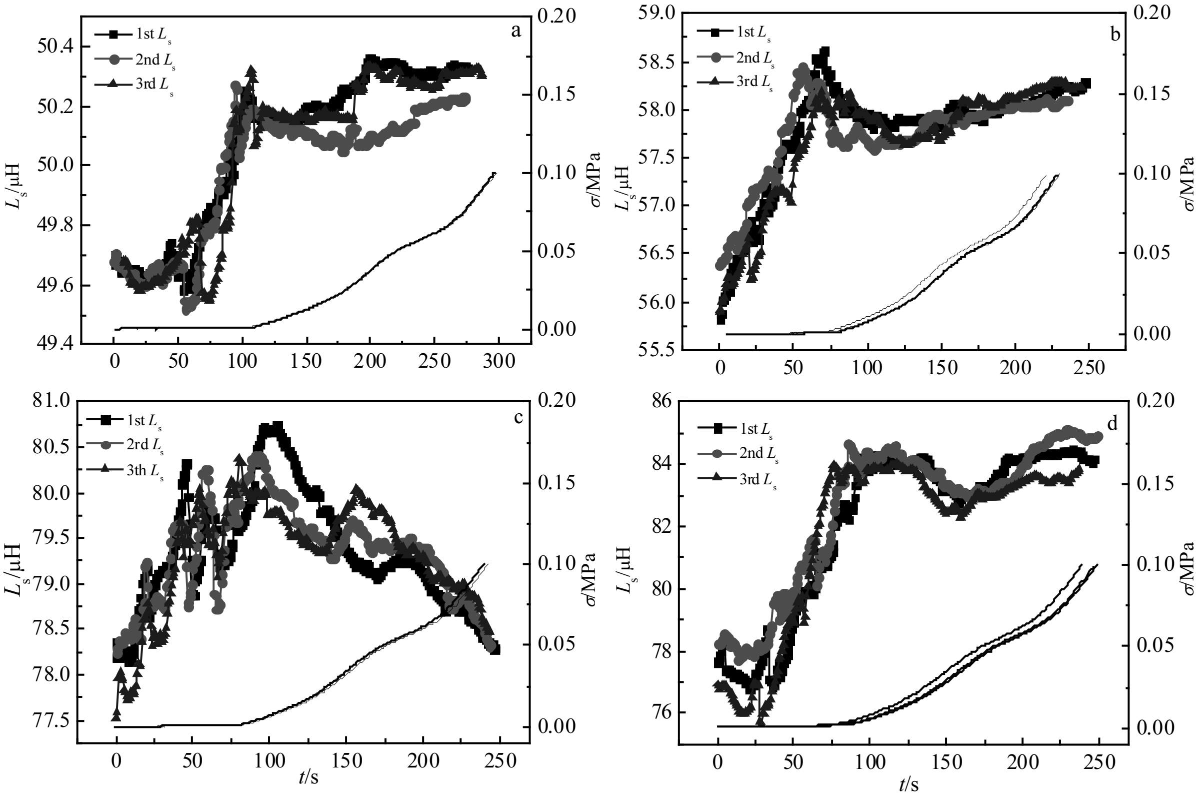

Fig. 10 Phenomenon of piezomagnetic property of $Fe_{73.5}Cu_1Nb_3Si_{13.5}B_9$ ribbons with different thickness: (a) 18~21 μm, (b) 24~28 μm, (c) 31~33 μm, and (d) 33~36 μm

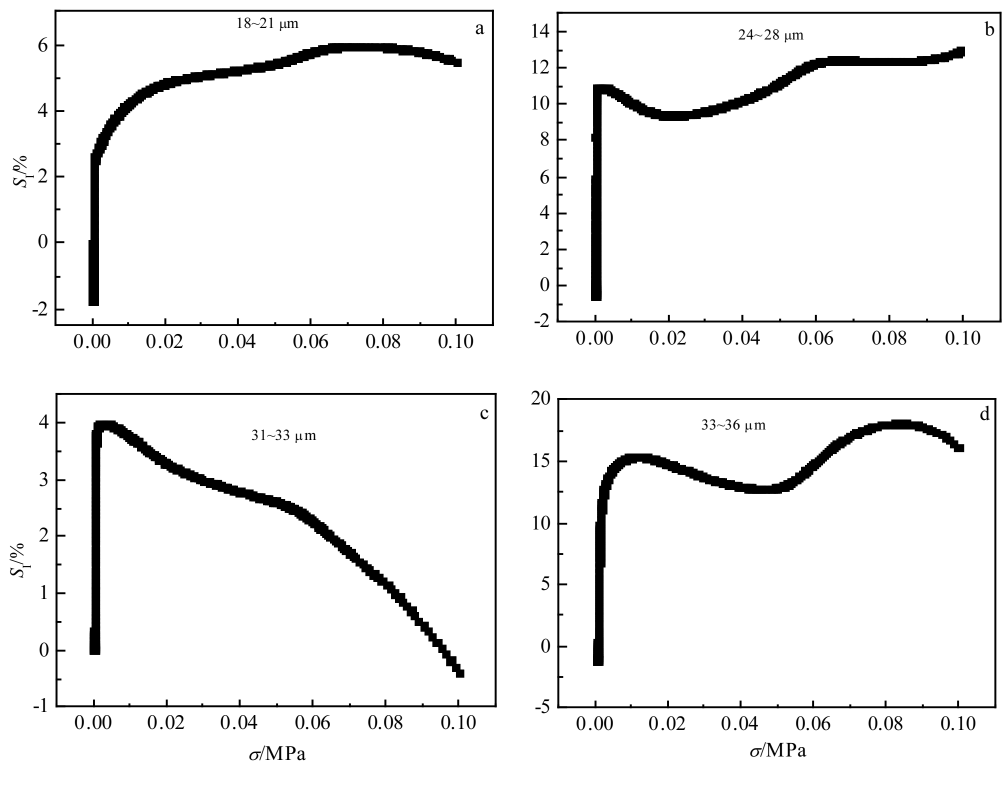

Fig. 11 Piezomagnetic property of $Fe_{73.5}Cu_1Nb_3Si_{13.5}B_9$ ribbon with different thickness: (a) 18~21 μm, (b) 24~28 μm, (c) 31~33 μm, and (d) 33~36 μm

variation: at the beginning of loading, the inductance shows a rapid rising, i.e. the sensitive range, after which, the inductance of the ribbon continue has small changes with the stress increasing, but the variation amplitude is much less than that in the week regime. The sample c with 31~33 μm is different, whose inductance value gradually drops with the pressure enhancing after the initial rise, which is called the negative piezomagnetic property. This specific mechanism remains to be further explored. The sensitive range of sample a~d determined by amplification method is 0~0.33, 0~0.35, 0~0.44 and 0~1.20 kPa, respectively. Comprehensive analyses of each sample situation of the piezomagnetic properties are listed in Table 1.

After investigating the influence induced by the thickness mentioned above, we then carried out some experiments to analyze the variations brought about by the width, and presented the results in Fig. 12. The width of testing sample a~d is 10, 20, 30 and 35 mm, respectively, with the thickness in the range of 24~28 μm. Our test results show that the strip initial inductance of ribbon is related to its size. The inductance increases gradually with width increasing, and the initial value of each sample is 677.58, 784.903, 829.039 and 863.457 μH, respectively. It is easy to find that the inductance change tendency of sample b, c and d is more or less the same: at the beginning of the loading, the stress imposed on the ribbons increases slowly, and then begins to increase sharply; however, the inductance presents a rapid increase in the initial loading process, and becomes much slower in the later section. The sample a has the same stress change tendency, while the change of inductance is different: at the beginning of loading, the inductance has a sharp increase, but it overall presents a downward trend after 80 s, which may be due to the uneven composition of the ribbon and slight differences resulting from the molding process.

Ribbons are different in small scale due to the thickness and the uneven internal stresses, otherwise, the external environment factors such as test temperature and air flow, also have influences on the results in the loading process. With the increase of the width, the inductance variation range increases obviously. We experimentally measure inductance rangeability in closed loop inductance of four kinds of ribbon is 56.206，167.829，75.744，98.813 μH, respectively.

Table 1 Summary of the test results with different thickness

Samples	$L_s(0)$/μH	L_s/μH	$S_{I\text{-}Max}$/%	P/kPa
a	9.75975	0.7721	7.8	0~0.33
b	16.21153	2.4146	13.5	0~0.35
c	38.39962	2.5359	4.12	0~0.44
d	38.39962	7.8043	19.8	0~1.20

Note: $L_s(0)$-inductance initial value, L_s-inductance rangeability, $S_{I\text{-}Max}$-piezomagnetic effect (absolute value), and P-pressure enhancing (sensitive area)

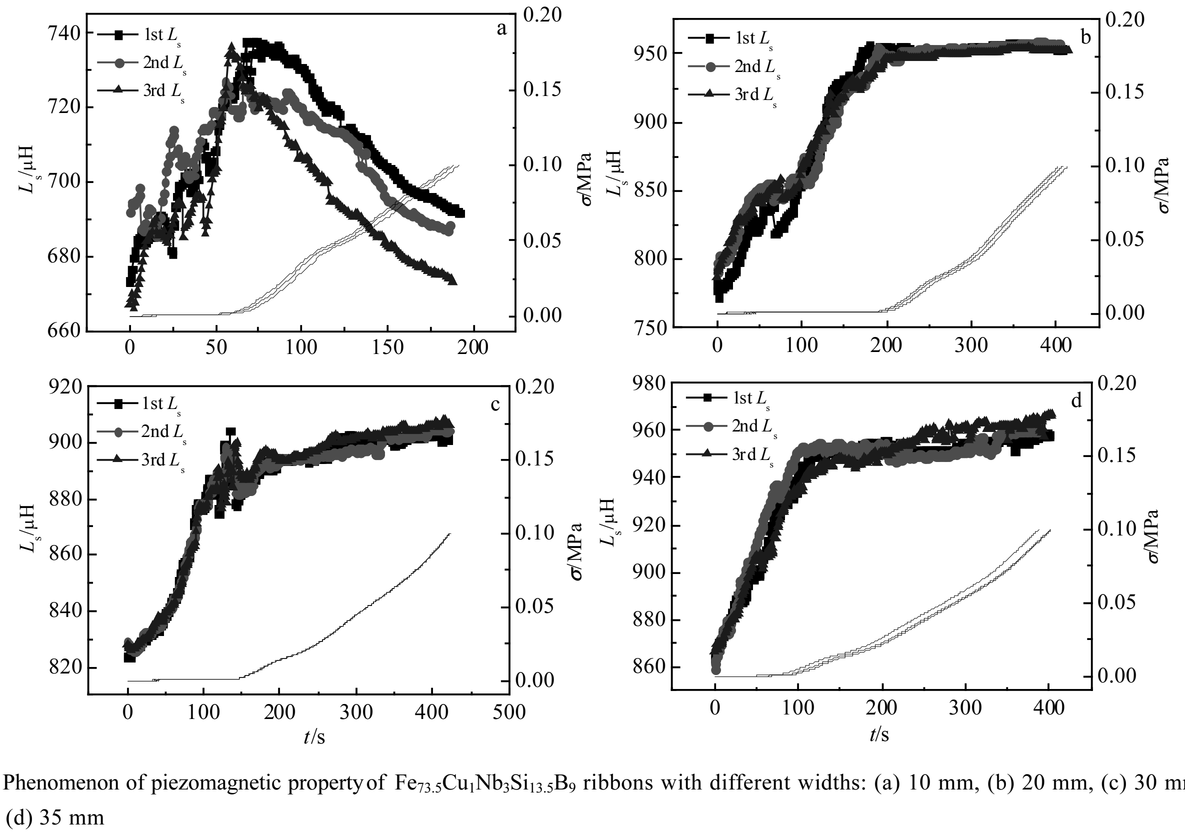

Fig. 12 Phenomenon of piezomagnetic property of $Fe_{73.5}Cu_1Nb_3Si_{13.5}B_9$ ribbons with different widths: (a) 10 mm, (b) 20 mm, (c) 30 mm, and (d) 35 mm

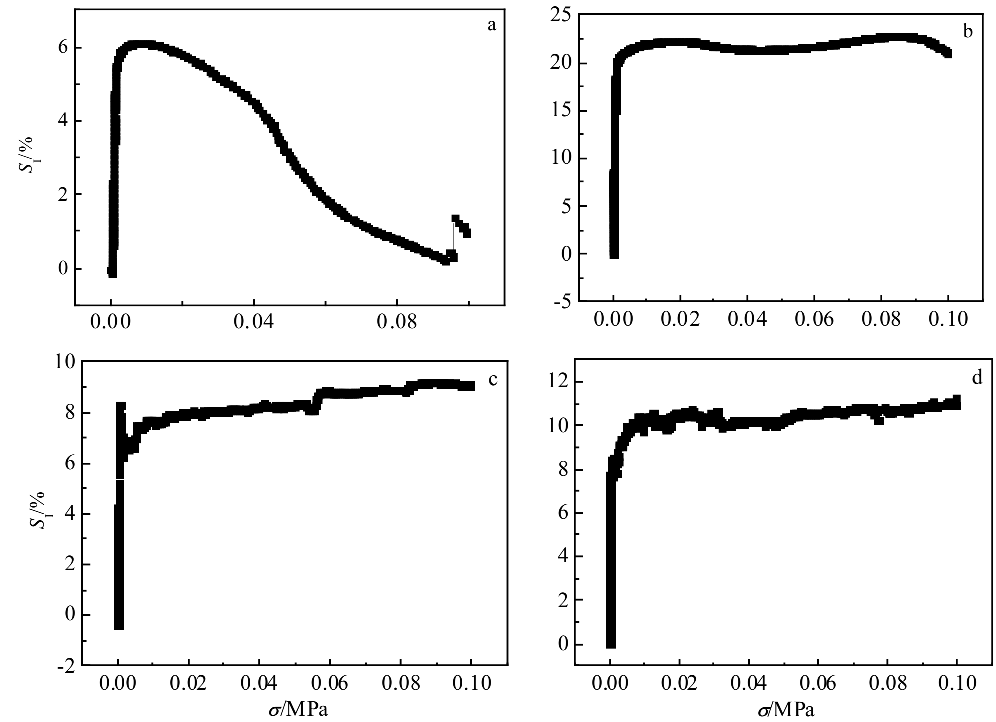

Fig. 13 Piezomagnetic property of $Fe_{73.5}Cu_1Nb_3Si_{13.5}B_9$ ribbons with different widths: (a) 10 mm, (b) 20 mm, (c) 30 mm, and (d) 35 mm

Fig.13 shows the curves of piezomagnetic property of $Fe_{73.5}Cu_1Nb_3Si_{13.5}B_9$ ribbons with different widths. The results are similar to those of the ribbons with different thickness, the inductance value of each group all have a rapid rising trend at the initial loading stage, the sensitive range is 1.20, 0.88, 0.66 and 0.16 kPa. Note that, the samples a with the width of 10 mm also presents a negative magnetic effect. After the sensitive range, the inductance shows a gentle decrease as the pressure increases gradually. It indicates that the piezomagnetic properties of the ribbons substantially increase with the rising of the width. The sample b has better piezomagnetic property than others, the absolute value of $S_{I\text{-}Max}$ reaches up to 22.02%, and $S_{I\text{-}Max}$ values of the remaining three groups samples are 6.18%, 9.17% and 11.23%, respectively. Comprehensive analysis of each sample situation of the piezomagnetic properties is shown in Table 2.

Table 2 Summary of the test results with different widths

Sample	$L_s(0)$/μH	L_s/μH	$S_{I\text{-}Max}$/%	P/kPa
a	677.580	56.206	6.18	1.20
b	784.903	167.829	22.02	0.88
c	829.039	75.744	9.17	0.66
d	863.457	98.813	11.23	0.16

Note: $L_s(0)$-inductance initial value, L_s-inductance rangeability, $S_{I\text{-}Max}$-piezomagnetic effect (absolute value), and P-pressure enhancing (sensitive area)

3 Conclusions

1) The piezomagnetic property of Fe-based amorphous alloy ribbons can be characterized stably and accurately by the improved inductance measurement device; the results obtained by this method are intuitive and accurate to describe the piezomagnetic property of the magnetic ribbons.

2) When the test frequency is 1 kHz, the voltage is 0.3 V and the compressive stress $\sigma \leq 0.1$ MPa, the amorphous magnetic material of $Fe_{73.5}Cu_1Nb_3Si_{13.5}B_9$ shows excellent piezomagnetic property, and the $Fe_{73.5}Cu_1Nb_3Si_{13.5}B_9$ amorphous ribbons of different specifications perform pretty good stability and repeatability after numerous repetitions. The ribbons are extremely sensitive to tiny stress. There is a rapidly rising trend of the inductance value in the initial pressure loading process. Moreover, the further amplifying experiment indicates that the sensitivity range is 0~1.5 kPa.

3) The size of $Fe_{73.5}Cu_1Nb_3Si_{13.5}B_9$ amorphous ribbon has a great influence on its piezomagnetic property. The initial inductance and its corresponding $S_{I\text{-}Max}$ value of the ribbon increase substantially along with the sample size increasing; with the same width, the ribbons with 33~36 μm in thickness exhibit superior piezomagnetic property to others, whose $S_{I\text{-}Max}$ is as high as 19.8%. While, with the same thickness, the ribbon with width of 20 mm shows an excellent piezomagnetic property among others and its $S_{I\text{-}Max}$ reaches 22.02%.

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铁基非晶合金带材压磁现象测试与表征方法改进及其压磁特性

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摘 要：在无接触式电感方法基础上自主开发改进了一套新的测试系统，改进的测量装置可以表征铁基非晶合金带材的压磁特性。研究表明，在 $\sigma \leq 0.1$ MPa 下，$Fe_{73.5}Cu_1Nb_3Si_{13.5}B_9$ 非晶带材具有良好的压磁特性，在测试电压为 0.3 V 、f=1 kHz 时，$Fe_{73.5}Cu_1Nb_3Si_{13.5}B_9$ 非晶带材均在多次试验中表现出很好的稳定性和重复性。带材对微小应力极为敏感，在对带材进行初步施压时，样品的电感值出现迅速上升趋势，通过放大实验，精确测到带材的应力敏感区间在 0~1.5 kPa 之间；不同厚度和宽度的 $Fe_{73.5}Cu_1Nb_3Si_{13.5}B_9$ 非晶带材对其压磁性能具有较大影响，测量的电感初值及其相应的 $S_{\text{I-Max}}$ 值总体上随着相应尺寸的增大而增大；带材宽度一定时，33~36 μm 厚带材的压磁性能最优，相应的 $S_{\text{I-Max}}$ 为 19.8%；带材厚度一定时，20 mm 宽的带材具有更加优异的压磁性能，其 $S_{\text{I-Max}}$ 可达 22.02%。

关键词：非晶合金；压磁效应；力敏特性；传感元件

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