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TOPICAL REVIEW — 110th Anniversary of Lanzhou University

Techniques of microwave permeability characterization for thin films*

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We review the microwave methods to characterize the material properties, including the established and the emerging techniques in material characterization, especially the permeability spectra of the magnetic thin films. Almost all aspects of the microwave techniques for characterizing the permeability of thin films at microwave frequencies, including the new methods developed by our group, are presented. Firstly, the introduction part is presented. Secondly, the coaxial-line with transmission/reflection methods and the pickup coil with electromagnetic induction method are presented. Thirdly, the most widely used shorted microstrip technique is discussed in detail by the equivalent circuit method, transmission line method, and electromagnetic induction method. Fourthly, the coplanar waveguide method and the near-field probe method are also introduced. Finally, the high temperature permeability characterization by using the shorted microstrip line, the near-field microwave probe, and the shorted microstrip line probe are described in detail. This paper may be useful for researchers or engineers who will build up such measurement fixture to make full use of the existing methods or to develop original methods to meet the requirements for ever-rising measurements.

Keywords: high frequency permeability, coaxial-line methods, induction coil method, shorted microstrip technique, coplanar waveguide method

PACS: 75.40.Gb, 84.40.Az, 76.50.+g **DOI:** 10.1088/1674-1056/ab3a8a

1. Introduction

Up to now, magnetic materials have been used in transformers, generators and motors, communication equipment, aerospace equipment, military equipment, high-density magnetic storage, *etc*.[1–5] In particular, with the rapid growth of information transmission frequency and processing speed, the communication frequency has been improved from MHz to GHz. This also puts forward a new development direction of applying the magnetic materials to microwave electronic devices, namely implementing high-frequency, miniaturization, and integration. As the working frequency of components increases, corresponding requirements are put forward for the high-frequency performances of soft magnetic materials, especially the high-frequency, higher permeability, and higher resonance frequency of soft magnetic thin film materials. Therefore, the accurate characterization of high frequency permeability of soft magnetic films is one of the most important issues to be developed in this field.

Many studies have been conducted on the methods of measuring high-frequency magnetic permeability of soft magnetic film, which can be roughly divided into the following methods according to the different measurement clamps used in the test: coaxial-line method, induction coil method, traditional shorted circuit microstrip test method, coplanar waveguide method, and near-field microwave microscope. In addition, according to the short-circuit microstrip line method, our group proposed an electromagnetic induction magnetic test method without calibration. These measurement methods are described in detail in the following sections.[6–14]

Moreover, microwave devices can be operated at temperatures higher than room temperature because of their thermal effects.[15–18] So, it is very necessary to investigate the magnetic properties of magnetic thin films at higher temperatures. However, the most common method of measuring the high frequency permeability of magnetic thin film at room temperature is to insert the thin film into the microwave fixture or to contact the microwave board.[19–27] These limitations mean that heating a sample is bound by the temperature tolerance of connecting wires and switching heads. These factors could limit the development of high temperature testing method. Therefore, only a few of studies about high-temperature permeability measurements, especially, of thin films were carried out.[7,29] Then a near-field microwave microscope (NFMM)

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method was developed to further increase the test temperature, which is a non-contact method.[7] The measurement technology was a big breakthrough because there is a little interspace between the microwave tip and the film surface, specifically, less than 20 μm, in order to obtain a significant signal. But the distance is too small, so the highest temperature of the NFMM method can only reach 423 K. Later, our group adopted the newly designed short-circuit microstrip line probe, which is also a non-contact test method, to further increase the test distance from sample to the test probe and further raise the test temperature to 473 K.[30] The above test methods will be described in detail in the following sections.

2. Coaxial-line method

Coaxial line method is a common method to measure the permeability of magnetic materials. This method is often used to test the samples of the powder compression ring, but the test of the thin film is less.[31] As early as around 1994, Acher *et al.* from CEA research center in France proposed to use coaxial line as a clamp to measure the complex permeability of magnetic films, as shown in Fig. 1.[32–34] The sample needs to be placed in the gap between the central signal line of the coaxial line and the outer ground wall. Since the electromagnetic wave transmitted in the coaxial line is the standard transverse electromagnetic field (TEM) mode, the complex permeability and dielectric constant of the dielectric material can be obtained from a relatively simple mathematical formula. Theoretical and experimental permeability spectra of CoNbZr thin film by coaxial line method are shown in Fig. 2. But the soft magnetic thin film sample is required to be deposited on a soft substrate so that the sample can be bent into a coil and inserted into the coaxial line. This is bound to introduce an additional stress into the film, making it difficult to extract the high-frequency magnetic spectrum of the soft magnetic film. Therefore, this method is generally only used to measure the high frequency complex permeability of the sample pressed into a ring, but is not applicable to testing the soft magnetic film deposited on a hard substrate.

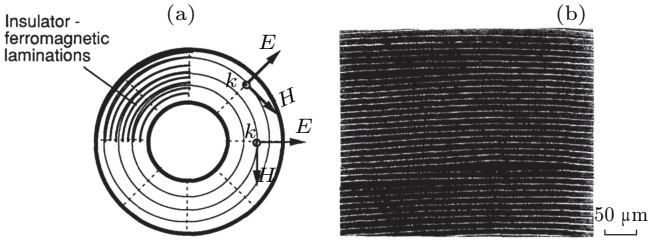


Fig. 1. (a) Sketch of geometry of laminated insulator-ferromagnetic on edge composite adapted to coaxial line geometry. Propagation vector $\boldsymbol{k}$, and $\boldsymbol{E}$ and $\boldsymbol{H}$ fields corresponding to fundamental mode are also indicated. (b) Micrograph of part of surface of a wound torus that is made by winding 0.4-μm-thick amorphous cobalt-based alloy deposited by magnetron sputtering on 8-μm-thick mylar.[30]

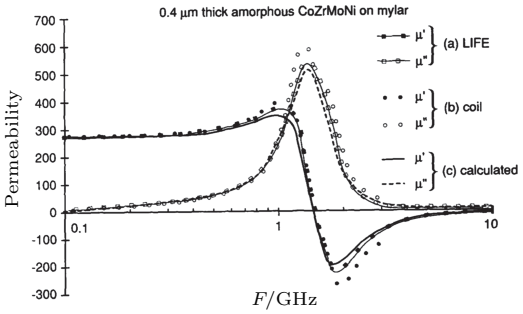


Fig. 2. Theoretical and experimental permeability spectra of CoNbZr thin film by coaxial line method.[30]

3. Induction coil method

In the 1990s, Yamaguchi *et al.* first proposed that the induction coil method can be used for measuring the high-frequency magnetic spectra of soft magnetic films. In a few years that followed, they improved the test equipment as shown in Fig. 3.[35–38] A driver coil (Drive Coil) was used to induce microwave excitation, the induction coil is a ring coil enclosed by a ribbon transmission line. Meanwhile, the induction coil (Pickup Coil) was used to detect voltage signal produced by electromagnetic induction. The film sample was placed inside the fixture. When measuring the high-frequency permeability of the soft magnetic film, the film sample is put into the loop of the induction coil, and the magnetic spectrum of the soft magnetic film sample is determined by measuring the electrical signal of the induction coil, which can be expressed as

$$\mu_{\mathrm{r}} t_{\mathrm{m}} = \frac{S_{\mathrm{c}}}{d_{\mathrm{m}}} \left(\frac{V_{\mathrm{s}} (Z_{\mathrm{s}} + 50)}{V_{\mathrm{r}} (Z_{\mathrm{r}} + 50)} - 1 \right) + t_{\mathrm{m}}, \tag{1}$$

where μ_{r} is the relative permeability of the thin film, t_{m} and d_{m} respectively represent the thickness and width of the thin film; S_{c} refers to the area of the induction coil; V_{s} and V_{r} respectively are the induced voltage when the sample is loaded and the induced voltage when the soft magnetic thin film sample is magnetized and saturated; Z_{s} and Z_{r} represent the impedance when loading the sample and the impedance after saturating the soft magnetic film sample. The test results from this method are shown in Fig. 4. It can be seen that the magnetic spectrum obtained by this method has a relatively flat real part value in the low frequency band, so that a relatively accurate initial permeability can be obtained. This is also an advantage of this method. However, with the increase of frequency, the measurement at higher frequency is limited due to the stray capacitance between the two coils, so the frequency of this test fixture reaches only up to 3.5 GHz. Therefore, the induction coil method is more suitable for measuring the complex magnetic permeability of soft magnetic film samples or ferrite thin film materials with resonance frequency less than 3 GHz.

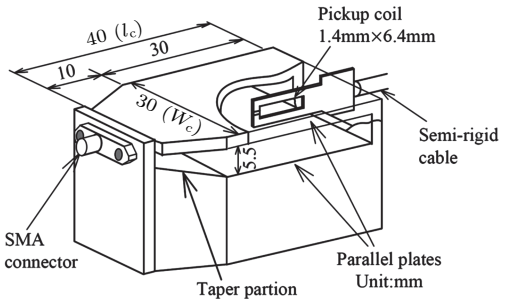


Fig. 3. Jig for pickup coil method.[36]

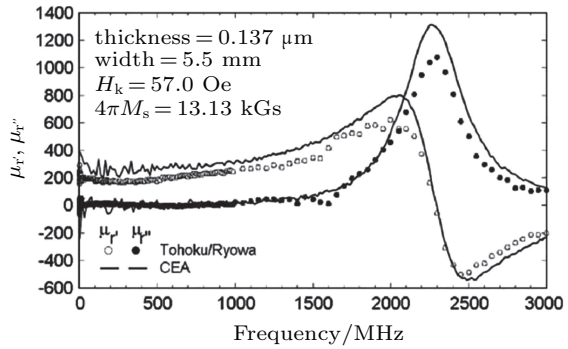


Fig. 4. Frequency profile of cross-measured permeability ($\mu_{r'}$ and $\mu_{r''}$) about CoNbZr thin film.[36] The units: 1 Oe = 79.5775 A·m^{-1}, 1 Gs = 10^{-4} T.

4. Traditional shorted circuit microstrip test method

Microstrip line is a microwave transmission line composed of a single conductor band supported on a dielectric substrate with high dielectric constant and low microwave loss. It is suitable for making planar transmission line of microwave integrated circuit. Accompanied by the development of microwave integrated circuits and low-loss dielectric materials, microstrip lines have been widely used in microwave integrated circuit and high speed pulse circuit. Its advantages include wide frequency band, small size, light weight, high reliability, easy connection with solid components, and easy integration of microwave components. Its disadvantages are large loss, difficult adjustment, only small and medium power applications. The dielectric loss of dielectric material should be very small in microstrip line. When measuring the high-frequency response of soft magnetic thin film materials with microstrip lines, soft magnetic thin film materials are generally placed between the upper and lower conduction bands as a medium, or soft magnetic thin film samples are placed above the microstrip lines.[39–41]

The conductor should have high conductivity, good stability, and good adhesion to substrate. It is widely used.

At present, equivalent circuit method, transmission line method and electromagnetic induction magnetic spectrum test method are the main methods to measure the magnetic spectrum of soft magnetic thin film with shorted circuit microstrip line. The disadvantage of this equivalent circuit method is that the highest test frequency can only reach about 6 GHz.[41–44] The transmission line method is the most widely used method at present.[37,38] Combining with traditional testing methods, our group proposed a soft magnetic thin film magnetic spectrum testing method without calibration based on electromagnetic induction principle.[11,12]

4.1. Equivalent circuit method

In 1999, Acher *et al.* used an equivalent circuit to analyze the short circuit microstrip line to obtain the magnetic spectrum of the soft magnetic film[45] as shown in Fig. 5. The device is based on a gold plated single coil which is equivalent to a microstrip line with width $W = 9$ mm. This coil was designed with $Z = 50\ \Omega$ line characteristic impedance and inner height $t_c = 1.70$ mm.[46,47] The reflection coefficient S_{II} of the coil is measured by using a network analyzer. Corresponding to the beginning of the microstrip line, the exact position about the phase reference plane (II) has to be determined. Filling the microstrip line with high dielectric materials ensures the electrical conductivity. The impedance Z_m is then obtained simply from

$$Z_m = Z_c \frac{1 + S_{II}}{1 - S_{II}}, \tag{2}$$

where Z_c represents the measured characteristic impedance.

The impedance measurement is carried out in two steps. The first step is to test only the substrate, and the second step is to test the ferromagnetic thin film. The permeability value is analyzed by the magnetic flux perturbation which is induced by the insertion of thin film into the coil.[48] The relative permeability is given by

$$\mu_r = 1 + \frac{\Delta Z}{i\mu_0 K t_s 2\pi f}, \tag{3}$$

$$\Delta Z = i2\pi f \mu_0 K (\mu_r - 1) t_s, \tag{4}$$

where μ is the permeability of the free space, K an unknown numerical constant which will be determined with a known sample, μ_r the relative permeability of the sample along the coil axis, t_s the sample thickness, and f the frequency of h. In this method the three basic elements of resistance R, inductance L, and capacitance C are used to equalize the microstrip line, and the value of each element can be obtained through the port impedance parameter of the microstrip line of the cavity.

In this study, CoFeNiMoSiB ferromagnetic film is tested. As shown in Fig. 6, the permeability spectrum is obtained by using this single adjustable parameter for each model. The theoretical permeability shows to be excellent agreement with the permeability of broad band.

As the test microwave frequency increases, the electromagnetic field distribution in the short circuit microstrip line will also become complicated, so the equivalent circuit also needs to be complicated, and more basic elements are needed

to be added to the appropriate position.[49–51] Therefore, the disadvantage of this equivalent circuit method is that the highest test frequency can only reach 6 GHz.

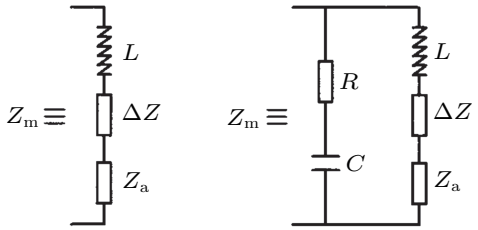


Fig. 5. Equivalent electrical circuit of (a) L model and (b) *RLC* model.[39]

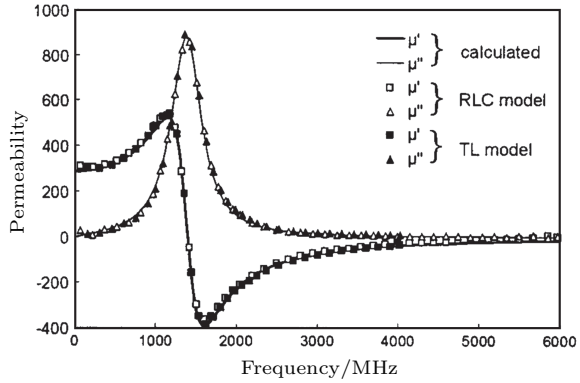


Fig. 6. Theoretical and experimental permeability spectrum of CoFeN-iMoSiB thin film.[39]

4.2. Transmission line theory

4.2.1. Three-step approach

In 2004, Seemann *et al.* analyzed the test of short circuit microstrip clamp based on transmission line theory. They used a broad-band technique to measure the high-frequency spectral complex permeability through a new theoretical approach and appropriate data processing procedure.[52] The method can be named "three-step" measurement method. The effective permeability is deduced from the measured strip line reflection coefficients respectively with and without a sample under test. For a lossless transmission line, the coefficient is associated with the effective relative permeability $\mu_{\rm eff}$ and the effective relative dielectric constant $\varepsilon_{\rm eff}$ of the transmission line, which can be expressed as

$$\gamma = \frac{{\rm i}\omega\sqrt{\mu_{\rm eff}\varepsilon_{\rm eff}}}{c_0}, \tag{5}$$

where $c_0 = \left(1/\sqrt{\varepsilon_0\mu_0}\right)$ represents the speed of light, which is related to vacuum permeability and vacuum dielectric constant. When different media are placed into the short-circuit microstrip line, its propagation coefficient will change. And the change will also be obtained through the reflection parameter of the port, which is denoted as S_{11}. As shown in Fig. 7, in the actual measurement process, we can divide the microstrip line into three parts, including the film sample part and the no-load part at both ends. The length and propagation coefficient of each part have been marked in the figure. Therefore, the S_{11} parameter of the port can be expressed as

$$S_{11} = -{\rm e}^{-2(\gamma_1 l_1 + \gamma_2 l_2 + \gamma_3 l_3)}. \tag{6}$$

The testing process is divided into three steps. The first step is to measure the port reflection parameter $S_{11}^{\rm empty}$ of the no-load short-circuit microstrip line; the second step is to measure the port reflection parameter $S_{11}^{\rm sub}$ of the short circuit microstrip line placed into the sample substrate; the third step is to measure the port reflection parameter $S_{11}^{\rm film}$ of the short circuit microstrip line placed into the soft magnetic film and its substrate. If the soft magnetic film sample and its substrate are square with the side length of $l_{\rm film}$, the length of the air part on each side of the sample is set to be $l_{\rm empty}$, then the total length of the microstrip line is $l = l_{\rm film} + l_{\rm empty}$. Therefore, the S_{11} parameter obtained from the three-step method can be expressed as

$$S_{11}^{\rm empty} = -{\rm e}^{-2\gamma_{\rm empty}\left(l_{\rm empty}+l_{\rm film}\right)}, \tag{7}$$

$$S_{11}^{\rm sub} = -{\rm e}^{-2\left(\gamma_{\rm empty}l_{\rm empty}+\gamma_{\rm sub}l_{\rm film}\right)}, \tag{8}$$

$$S_{11}^{\rm film} = -{\rm e}^{-2\left(\gamma_{\rm empty}l_{\rm empty}+\gamma_{\rm film}l_{\rm film}\right)}, \tag{9}$$

where $\gamma_{\rm empty}$, $\gamma_{\rm sub}$, and $\gamma_{\rm film}$ represent the propagation coefficients of the microstrip line when the air part, only the substrate and the soft magnetic film with substrate are respectively placd in the fixture. Take the logarithms of both sides of the above three expressions, and calculate the resulting expressions by the substitution method to obtain $\gamma_{\rm sub}$ and $\gamma_{\rm film}$, respectively, as shown in the following equations:

$$\gamma_{\rm sub} = \frac{l_0 \ln\left(-S_{11}^{\rm sub}\right) - l_{\rm empty}\ln\left(-S_{11}^{\rm empty}\right)}{-2l_{\rm film}l_0}, \tag{10}$$

$$\gamma_{\rm film} = \frac{l_0 \ln\left(-S_{11}^{\rm film}\right) - l_{\rm empty}\ln\left(-S_{11}^{\rm empty}\right)}{-2l_{\rm film}l_0}. \tag{11}$$

Since no magnetic signal exists when only the substrate is placed, the effective permeability in the propagation coefficient is equal to 1. Since the soft magnetic film is very thin, if its influence on effective dielectric parameters is ignored, according to Eq. (5), the effective permeability of the soft magnetic film can be expressed as follows:

$$\begin{aligned}\mu_{\rm eff} &= \left(\frac{\gamma_{\rm film}}{\gamma_{\rm sub}}\right)^2 \\ &= \left(\frac{l_0 \ln\left(-S_{11}^{\rm film}\right) - l_{\rm empty}\ln\left(-S_{11}^{\rm empty}\right)}{l_0 \ln\left(-S_{11}^{\rm sub}\right) - l_{\rm empty}\ln\left(-S_{11}^{\rm empty}\right)}\right)^2.\end{aligned} \tag{12}$$

The above equation gives the effective relative permeability, that is, the average effect of the permeability of soft magnetic film on the entire microstrip line. The relative permeability is calculated from the relative effective permeability by applying the following relation:

$$\mu_{\rm r} = \frac{\mu_{\rm eff} - 1}{K(d/h)}, \tag{13}$$

where d is the thickness of the soft magnetic film, h is the height of the short circuit microstrip line, and K is a dimensionless scaling factor, which is derived by adjusting the measured frequency-dependent permeability to the initial permeability. As shown in Fig. 8, the method is suitable for measuring the permeability of thin magnetic FeCoAlN film with uniaxial anisotropy. But some deviation appears when the frequency exceeds 2.2 GHz. Although the above-mentioned three-step method can obtain the complex permeability of the soft magnetic film, the influence of the dielectric constant of the soft magnetic film is ignored. In addition, the size and position of the soft magnetic film sample placed cannot be completely consistent with those of the substrate. Therefore, the above three-step method still has many shortcomings.

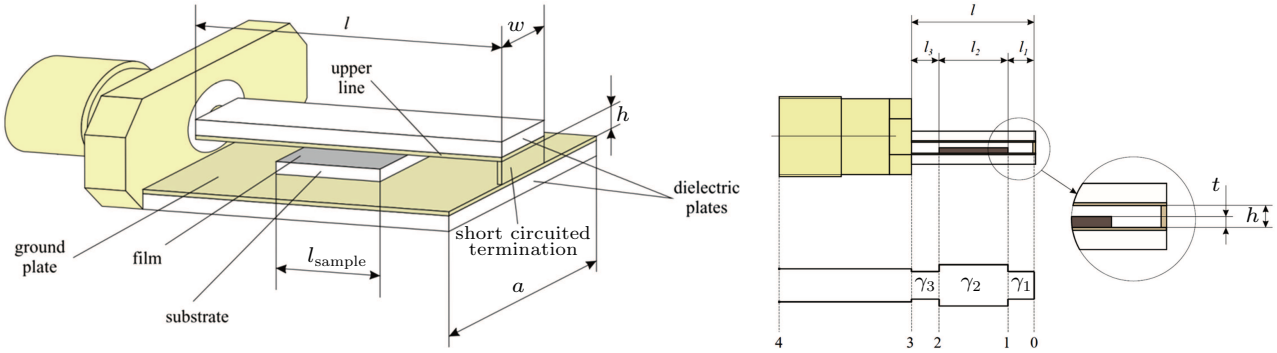


Fig. 7. Design of the short-circuited strip line.[50]

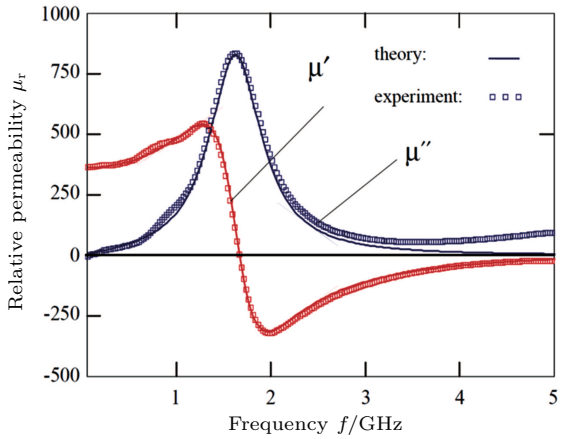


Fig. 8. Theoretical and experimental relative permeability of FeCoAlN film.[50]

4.2.2. Four-step method

Based on the above three-step measurement process, the "four-step" measurement method is proposed to make up for the deficiencies in the above measurement process. This measurement method covers four steps. The first step is to measure the port reflection parameter S_{11}^{emp} of the no-load short circuit microstrip line. The second step is to measure the port reflection parameter $S_{11}^{\text{emp-mag}}$ of the cavity short circuit microstrip line clamp when the external magnetic field is strengthened. The strong magnetic field is much stronger than the saturation field of the sample. The third step is to measure the port reflection parameter S_{11}^{film} of the short circuit microstrip line when the soft magnetic film sample is placed into the fixture. The fourth step is to measure the port reflection parameter $S_{11}^{\text{film-mag}}$ of the short circuit microstrip line clamp with soft magnetic film samples when the external magnetic field is strengthened. The size of the strong magnetic field is the same as that in the second step. In the third and fourth step, soft magnetic film samples are placed into the fixture. The transmission coefficient of the transmission line containing the sample part can be respectively expressed as follows:

$$\gamma_{\text{film}} = \frac{\mathrm{i}2\pi f}{c_0}\sqrt{\mu_{\text{eff}}\varepsilon_{\text{eff}}},$$
$$\gamma_{\text{film-mag}} = \frac{\mathrm{i}2\pi f}{c_0}\sqrt{1\cdot\varepsilon_{\text{eff}}}. \tag{14}$$

The strong magnetic field added in the fourth step is much higher than the saturation field of the soft magnetic film, so the effective permeability is close to 1, or the magnetic moment is frozen. In addition, the dielectric parameters do not change with the addition of strong magnetic field. Therefore, the effective permeability of the transmission line containing the part of soft magnetic film can be obtained by dividing the above equation as shown in the following equation:

$$\mu_{\text{eff}} = \left(\frac{\gamma_{\text{film}}}{\gamma_{\text{film-mag}}}\right)^2. \tag{15}$$

Because in the fourth step of adding the strong magnetic field, the transmission line without the sample part is also affected by the strong magnetic field, so it is also necessary to measure the result in the case of short circuit in the cavity of the microstrip line when the external magnetic field is strengthened, which is the necessity of the second step of measurement. As in the previous three-step method, the first measurement is to subtract the background effect of the microstrip line. Thus, the effective permeability of the "four-step method" can be

deduced as follows:

$$\mu_{\text{eff}} = \left[\frac{l_0 \ln\left(-S_{11}^{\text{film}}\right) - l_{\text{emp}} \ln(-S_{11}^{\text{emp}})}{l_0 \ln\left(-S_{11}^{\text{film-mag}}\right) - l_{\text{emp}} \ln(-S_{11}^{\text{emp-mag}})} \right]^2 . \quad (16)$$

The data processing procedure of the high-frequency permeability of soft magnetic films, derived from the effective permeability, is the same as the "three-step method" mentioned above. The advantage of the "four-step method" proposed is that it does not need to consider the influence of the substrate nor to measure the S_{11} parameter of the sample substrate, thus avoiding the influence of the placement of the soft magnetic film sample on the measurement of the high-frequency permeability. This method is also one of the most commonly used high-frequency magnetic spectrum testing methods in our research group.

4.3. Electromagnetic induction magnetic test method[11,12]

A standard sample is required to calibrate the test results, thus yielding a calibration factor "K", in the process of measuring the magnetic spectrum of the soft magnetic film with a short circuit microstrip line clamp. Recently, some researches have shown that the scaling factor K varies with frequency, which can be obtained by simulation software or from the complex mathematical formula calculation,[53,54] but this process is relatively tedious. The main work of our working group is to study the calibration factor in the process of magnetic spectrum measurement and to propose a method of measuring the magnetic spectrum of soft magnetic film based on the principle of electromagnetic induction without calibration. The details will be given below.

4.3.1. Short circuit microstrip clamp

The main material of the whole test fixture is brass, and the ground pole and shielding cavity part of the fixture are made by metal processing technology as shown in Fig. 9, which can play a shielding and fixing role.[12] The core component is microstrip wire conductor, which is Rogers 5880 dielectric plate. Its dielectric constant is 2.2, and the tangent of electrical loss is 0.004. Then the microstrip wire and cable are connected through the SMA connector as shown in Fig. 9. The dielectric plate is used to fix the microstrip wire, which is not the dielectric material between the microstrip and the earth pole.

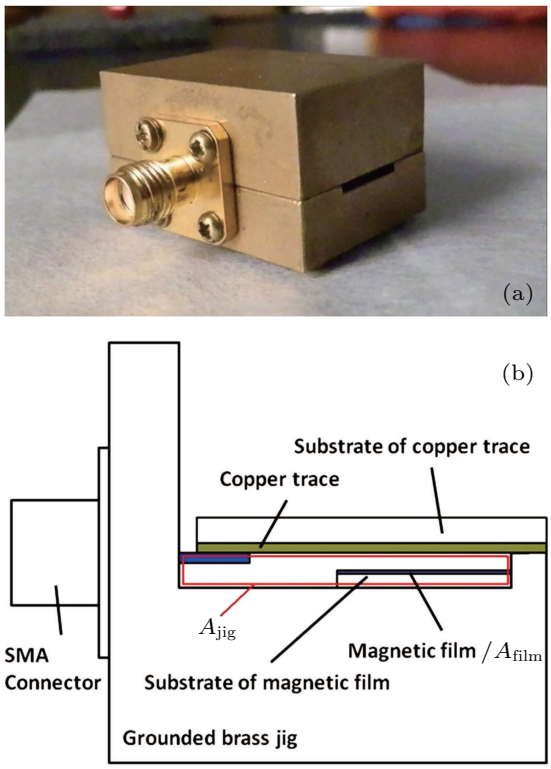


Fig. 9. (a) The short-circuit microstrip line fixture in the Magnetic Laboratory of Lanzhou University, and (b) the corresponding schematic diagram of panel (a).[12]

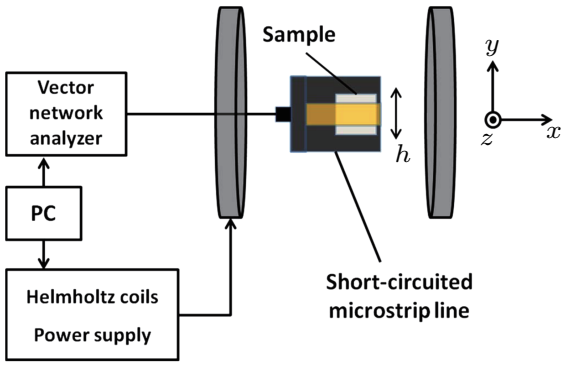


Fig. 10. The schematic diagram of the permeability measurement system.[12]

In the process of making short circuit microstrip line fixture, the most important thing is that the fixture is designed to have 50 Ω for impedance matching. The distance d between upper signal line and ground plate is 0.8 mm, which could provide enough space to place a magnetic thin film with the upper signal line width $w = 3.94$ mm and the fixture length $l = 9$ mm. The short circuit microstrip line fixture has many advantages, such as simple analysis with transmission of quasi-TEM wave, broad band frequency range, and higher sensitivity. So in many research institutions this test method is adopted popularly. The test fixture is connected to the VNA via a cable. The whole device controlled by a computer also needs to be equipped with Helmholtz coils to provide a static

magnetic field H_{app} along the microstrip line (x axis) during the test. The schematic diagram of the measurement system is shown in Fig. 10. The sample is placed at the end of the short-circuited microstrip line jig. In the testing process, the value of the microwave field $\boldsymbol{h}$ can be changed, which is perpendicular to the x axis. Then, more information can be obtained because complex permeability has different values in different applied magnetic fields.

4.3.2. Electromagnetic induction magnetic spectrum test method

In order to analyze the measurement results by an induction method, the short-circuit microstrip line is considered as a single coil, which means it serves as both a drive coil and an induction coil as shown in Fig. 10. The advantage of this analysis method is that we do not need to consider the influence of electrical signals on the measurement, and the change of magnetic flux is only related to the magnetic signal. According to the Ampere circuit law, a microwave field h induced by the driven coil can be obtained as follows:[55]

$$h = Ki/w, \tag{17}$$

where i represents the alternating current, and K is a dimensionless coefficient determined by the designed fixture. As is well known, the magnetic flux ϕ_{film} can be detected by pickup coil when inserting the magnetic thin film into the fixture. When a magnetic thin film is under a saturation field, the magnetic flux will be nearly zero. So, the change of the magnetic flux $\Delta\phi$ with and without the high magnetic field is obtained below

$$\phi_{film} = \mu_0(\mu_r - 1)h \cdot A_{film} + \mu_0 h \cdot A_{jig}, \tag{18}$$

$$\Delta\phi = \mu_0(\mu_r - 1)h \cdot A_{film}, \tag{19}$$

where μ represents the permeability of the free space, μ_r the relative susceptibility, A_{film} the sectional area of the magnetic film, and A_{jig} the sectional area of the microstrip line. At the input-port, according to the electromagnetic induction law, the change of impedance can be expressed as follows:

$$\begin{aligned}\Delta Z &= Z_{film} - Z_{filmmag} = \frac{1}{i} \cdot \frac{\mathrm{d}(\Delta\phi)}{\mathrm{d}t} \\ &= \frac{\mu_0(\mu_r - 1) \cdot A_{film} K}{w} \cdot (\mathrm{i}2\pi f),\end{aligned} \tag{20}$$

where f denotes the frequency of the microwave, Z_{film} the port impedance of the jig without the saturation field, $Z_{filmmag}$ the port impedance of the jig with the saturation field, and K the dimensionless coefficient, which can be obtained from the impedance Z_{emp} of the short-circuit microstrip line without film as follows:

$$K = \frac{Z_{emp}}{\mathrm{i}\mu_0 A_{jig}/w \cdot 2\pi f}. \tag{21}$$

Therefore, the relative permeability of the magnetic thin film can be given from Eqs. (20) and (21) as follows:

$$\mu_r = 1 + \frac{Z_{film} - Z_{filmmag} - Z_{emp} + Z_{empmag}}{Z_{emp}} \cdot \frac{A_{jig}}{A_{film}}, \tag{22}$$

where the port impedance Z can be obtained by the measured S_{11} parameter and expressed as

$$Z = Z_0 \frac{1 + S_{11}}{1 - S_{11}}, \tag{23}$$

where Z is the characteristic impedance and S_{11} the reflection coefficient which is measured by short-circuit microstrip line connected to the VNA.

From the above derivation, we know that the process of measuring magnetic spectrum by short circuit microstrip line will be completed without calibration, which is very meaningful for the accurate measurement of soft magnetic thin film magnetic spectrum. In order to achieve the purpose of measuring complex permeability, the values of thickness, length and width of the sample need to be measured. Meanwhile, four S_{11} parameters need to be obtained through four test steps. The measurement process is divided into four steps. The first step is to measure the S_{11} parameters of the cavity short-circuit microstrip clamp and calculate the port impedance Z_{emp}. The second step is to measure the S_{11} parameters of the cavity short-circuit microstrip clamp when the external magnetic field is strengthened, and calculate the port impedance Z_{empmag}. The third step is to measure the S_{11} parameters of the short-circuit microstrip line fixture when being placed in the soft magnetic film sample, and calculate the port impedance Z_{film}. The fourth step is to measure the S_{11} parameters of the short-circuit microstrip clamp when the external magnetic field is strengthened and the soft magnetic film sample is placed, and then calculate the port impedance $Z_{filmmag}$. Through these four steps the permeability of the film can be obtained.

In this study, FeNi and FeN films are selected as the research object, because of their good soft magnetic properties. It is shown that these samples have good uniaxial anisotropy tested by VSM as shown in Figs. 11(a) and 11(c). The complex permeability spectra of FeNi and FeN films are measured in frequency range from 100 MHz to 9 GHz respectively as shown in Fig. 11(b) and 11(d), by using this new designed fixture and this new method. In addition, the experimental values can fit well with the theoretical analytical curves obtained from the Landau–Lifshitz–Gilbert equation as shown in Figs. 11(b) and 11(d).[56]

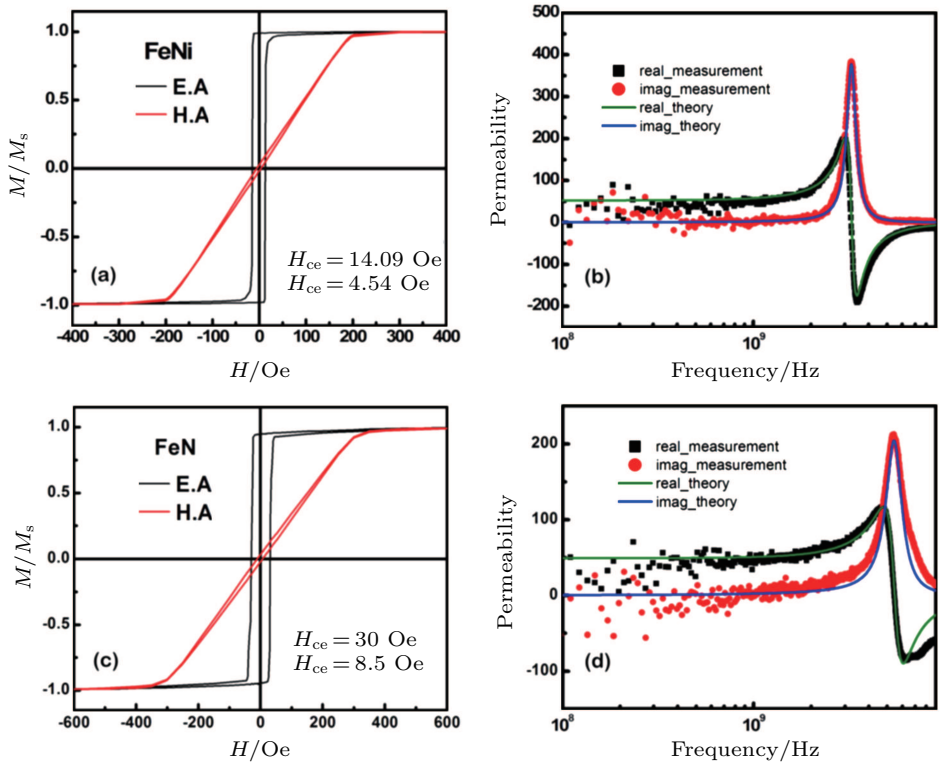


Fig. 11. (a) and (c) Hysteresis loops and (b) and (d) complex permeability spectra of FeNi and FeN films.[12]

5. Coplanar waveguide method

Coplanar waveguide is widely used as a high-frequency magnetic test fixture.[57–60] It is named coplanar waveguide because its signal line and ground wire are in the same plane. It has many advantages, such as simple structure, easy integration, and convenient-to-place samples. Samples can be freely rotating on the top of test fixture. The sample is placed on the coplanar waveguide (CPW) structure as indicated. The mutually orthogonal static applied field $\boldsymbol{H}_{\text{ext}}$ and the microwave field h are in the plane of the film. As shown in Fig. 13, it is a millimeter-sized CPW made on a radio frequency (rf) dielectric plate. The high-frequency magnetic spectrum results are obtained by using the equivalent circuit method. The complex permeability of the soft magnetic film can be derived from the equivalent circuit, which can be expressed as follows:

$$\mu = \frac{Z_0}{\mathrm{i}clt\mu_0\omega}\left(\frac{1+S_{11}^1-S_{21}^1}{1-S_{11}^1}-\frac{1+S_{11}^0-S_{21}^0}{1-S_{11}^0}\right), \quad (24)$$

In the formula, superscripts 1 and 2 of S refer to S parameters when loading the film sample and when saturating the sample with magnetization respectively, l and t denote the length and thickness of the soft magnetic thin film sample respectively, Z and ω represent the characteristic impedance of CPW and the microwave frequency respectively, c is a scaling factor, which is associated with the structure size of CPW. Generally, this test method needs to be calibrated by using a standard sample. Complex magnetic susceptibility of a CoFe film is extracted by the coplanar waveguide method as shown in Fig. 2 in Ref. [56].

Due to its structural advantages, the coplanar waveguides are also used to test the high-frequency magnetism of soft magnetic films at low temperatures as shown in Fig. 12.[61] It is a U-shape double-port coplanar waveguide, which is combined with other cryogenic devices to form a cryogenic high-frequency magnetic testing system. Shown in Fig. 12 is a simplified sketch of the configuration for measuring an external field generated by the split coils that are (b) parallel and (c) perpendicular to the sample plane, respectively. The dipper probe in the horizontal plane can be rotated to change from one configuration to the other. Another advantage of coplanar waveguide is that it can be combined with microwave probe stations to measure the miniaturized samples.[58] As shown in Fig. 13, CPW at a micron level is produced by micro-nano processing technology, and the soft magnetic film sample is deposited between the Gap of CPW by the nesting technology. The central conductor of the CPW is about 260-μm wide. High-frequency micro-probes and coaxial cables are used to connect the structure to the VNA. The sample's anisotropy axis (AA) makes an angle φ_H with respect to the direction of $\boldsymbol{H}$.

Therefore, the method of using coplanar waveguide transmission line to measure the complex permeability of the soft magnetic film has many advantages. Commercial cryogenic ferromagnetic resonance (FMR) uses this method, to be integrated into the physical property measurement system (PPMS) test equipment. But there are also many drawbacks. For example, the miniature CPW can only obtain the resonant peak

of the sample from the S parameter, and the calibration of the standard sample is required in the derivation of the complex permeability.

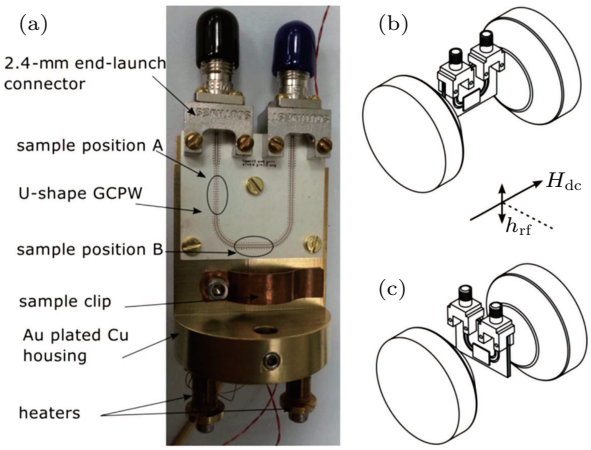


Fig. 12. Probe-head with U-shape GCPW.[59]

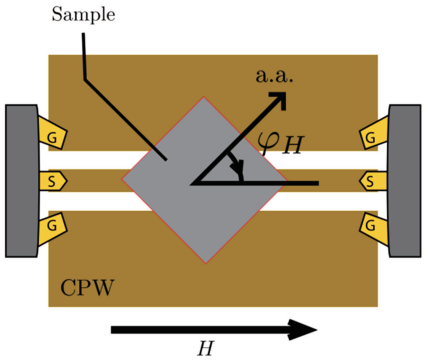


Fig. 13. Schematic diagram of coplanar waveguide (CPW) structure and sample placed on top of it.[58]

6. Near-field microwave microscope

Because the microwave probe has the advantage of low background interference and high signal-to-noise ratio, it is widely studied in high frequency microwave measurement. Lee *et al.* developed a technique to test the high-frequency permeability of thin film by the near-field microwave probe, in which used is a coaxial transmission line resonator terminated with a loop soldering the inner conductor to the outer conductor.[62] In this measurement method a small sample area is excited and the electromagnetic response is detected because of the loop behaving like an electrical short circuit. When the magnetic film is placed in the loop, the boundary conditions at the end of the resonator are changed.

Then, Mircea *et al.* improved the above technique.[63] The main improvement in their technology is directly connecting 25 μm thin lines to the internal and external coaxial lines, thereby reducing the distance between the probe and the sample and enhancing the coupling between the test circuit and the sample. Then they improved the method, that is, the 500-nm-thick copper film is deposited directly on the cross section of the coaxial cable, and the electroplated copper is made into a narrow bridge through a special process to replace a thin wire (25-μm in thickness) as depicted in Fig. 14. The corresponding alternating current in the copper "microloop" generates the rf magnetic field h_{rf}, which acts as an excitation field of the probe. At the same time, the loop can also be used as a signal acquisition coil to sense the response of samples to microwave field. The permeability is extracted from the measurement through the following relation:

$$\mu_r \approx k^2 \frac{L_X}{L_0} \frac{1}{\omega t_0 \mu_0} [\mathrm{Im}(\Delta Z) - \mathrm{i}\,\mathrm{Re}(\Delta Z)], \tag{25}$$

where μ_0 represents the free-space permeability, L_0 the inductance of probe and loop, t_0 the sample thickness ($t_0 <$ skin depth δ), k a dimenasionless coefficient ($0 \le k \le 1$) describing the probe-to-sample coupling, $k = \sqrt{M^2/(L_0 L_X)}$, with M being the mutual inductance between the probe and sample, and L_X the inductance of the probe's image in the sample ($L_X \to L_0$ for a perfect image).

To further verify the sensitivity of their probes, the materials of different thickness values from 300 nm to 15 nm are tested, which apparently allows the samples of different geometric shapes to be characterized. As shown in Fig. 4 in Ref. [61], the signal is, indeed, strong for samples as thin as 15 nm. In addition, the signal-to-noise ratio and spatial resolution can be further improved by reducing the probe size. However, the disadvantage of this method is that the measured high frequency magnetic spectrum cannot accurately give the initial permeability of soft magnetic film sample.

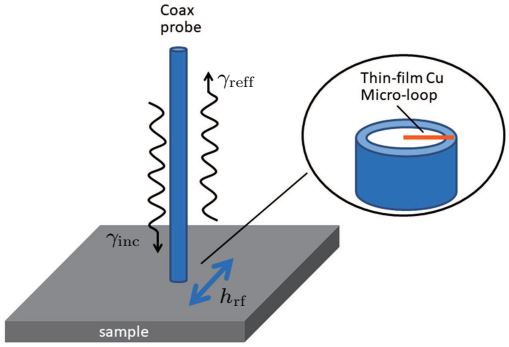


Fig. 14. Schematic diagram of FMR coax microloop probe and its equivalent circuit.[61]

7. High temperature measurement for thin film materials

Microwave components of high-frequency magnetic materials are widely used in various fields. With the development of microelectronic integrated circuit technology, the microwave components are gradually developing towards miniaturization and high-frequency, which requires that high-frequency magnetic materials should have higher permeability and cut-off frequency. The high-frequency soft magnetic film has a huge demagnetizing field in the direction of the

vertical film, so that the thin film material has a higher permeability under the condition of the same cut-off frequency, and it is expected to be used in the future microwave magnetic devices, such as microwave isolator, phase shift, circulator, filter, *etc*.[64–69] Obviously, the device in use inevitably will have a thermal effect, in addition to the high-frequency device itself, all kinds of surrounding electronic components will have the heating phenomenon. Besides, due to the change of ambient temperature, high-frequency magnetic devices are required to have stable high-frequency properties in a wide temperature range, and even some devices themselves work in an environment with large temperature fluctuations. For example, aviation equipment or field observation and detection equipment needs to operate in an environment with large temperature fluctuations outdoors or at high altitude. Therefore, the thermal stability of these active or passive high-frequency magnetic devices operating at high temperature determines the stability of the device performance. If the thermal stability of the material is not good, the performance of the material will be worse than that at a certain temperature point or even a phase change will occur, which will inevitably lead the overall performance of the device to change, and the whole equipment to fail to work or even to suffer unexpected dangers. Therefore, in the design process of high frequency magnetic devices, it is necessary to know the variation characteristics of core high frequency magnetic materials with temperature in advance.

Therefore, how to characterize the permeability of thin film materials in the GHz frequency band, especially at high temperature, has become an urgent problem to be solved. To evaluate the permeability of thin films, the most common method is that the sample is inserted into the microwave fixture or contacts the microwave board because of the limitation of the testing mechanism.[25,70–75,77,78] Thus, only several techniques have been developed for measuring the high-temperature permeability of magnetic thin films.[7,77] They will be described below.

7.1. Shorted microstrip transmission-line method

In 2003, Ledieu *et al.* published a paper on measuring ferromagnetic thin film microwave spectrum based on the fixture of short-circuit microstrip line, in which the short-circuit microstrip line is connected to the ground by brass short-circuit, and the other end is connected to the transmitter of SMA coaxial joint by welding technology.[79] In this way, the magnetic film needs to be pushed into the inside of the clamp of the microwave transmission line. By heating the whole device, the temperature can be accurately controlled in a range of 77 K–400 K and the frequency can reach 6 GHz. The device, shown in Fig. 15, is thickened at the ground end to allow it to be used for a heater. A heater is used to increase and control the temperature, and the bottom of the test fixture is cooled by soaking it in liquid nitrogen. The temperature probe is located below the sample. The testing principle of the device is based on standard single-coil perturbation technique.[28,80] The variation of permeability spectrum of ferromagnetic layer with ferromagnetic layer impedance is calculated by an *RLC* model. In this work, the high-frequency permeability spectra of CoNbZr films at different temperatures (90 K $\sim$ 390 K) and different frequencies (500 MHz $\sim$ 6 GHz) are measured as shown in Fig. 5 in Ref. [77].

However, the temperature of the test device is not too high and the temperature limitation is mainly due to the temperature limitation of the fixture parts, such as the substrate of printed circuit board (PCB) or the solder used to weld PCB and coaxial connectors. Therefore, the maximum temperature of the test device should be less than 150 °C. Obviously, it is necessary to develop a non-contact test method, that is, the sample does not need to contact the test fixture in measuring the high frequency permeability of the magnetic film at a higher temperature.

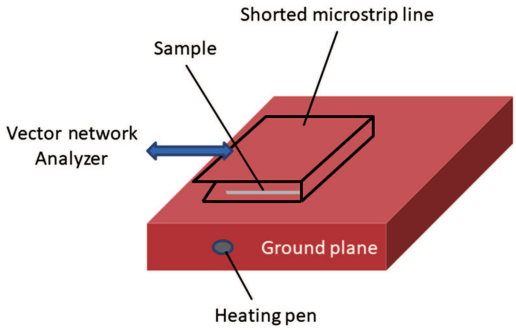


Fig. 15. The magnetic spectrum testing device in a wide range of temperatures.[77]

7.2. Near-field microwave microscope

In 2011, Hung *et al.* applied microwave near-field detection technology to thin-film microwave permeability tests at varying temperatures, and published a paper on a new test method that could measure temperatures up to 423 K by using specific near-field microwave probes to measure high-frequency magnetic characteristics up to 5 GHz.[7] The measurement fixture is shown in Fig. 16, which is a coaxial cable with short ends. Coaxial outer conductor and inner conductor are connected by 50-μm diameter gold wire through welding technology. Thus, a micro-loop is formed at the tip, generating an rf magnetic field when an electric current passes through it. The production process of the probe tip is shown in Fig. 16(a). The near-field microwave microscope (NFMM) probe is connected to the vector network analyzer through adapters and cable as shown in Fig. 16(b). The film sample is heated by a heater, and the z-axis position of the sample can be controlled through the Z-piezo. In order to improve the coupling strength between the sample and the test probe, the distance between

the micro circuit and the magnetic film must be small enough. In the test device, the distance is controlled to be 20 μm, so that the microcirculation will not directly contact the magnetic film surface.

However, in order to obtain an enough signal, the sample surface must be very close to the near-field probe, usually less than 20 μm, although it does not need to contact the top of the near field probe. This inevitably leads the test temperature of this measurement method to be unable to further increase, but to reach only 423 K.

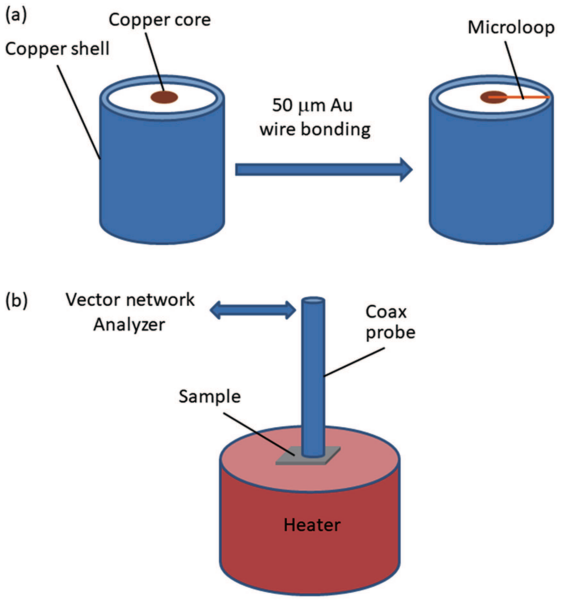


Fig. 16. (a) Microprobe manufacturing process, and (b) schematic diagram of the whole heating test system.[7]

7.3. Shorted microstrip line probe

By combining previous work, our working group has developed a non-contact method, that is, short circuit microstrip probe. The short circuit microstrip probe is modified from the traditional short circuit microstrip clamp.[81] The measuring principle is the same as that for the short-circuit microstrip transmission line method.[25] The probe structure diagram is shown in Figs. 17(a) and 17(b). The microstrip wire is on one side of the microwave PCB, the other side is completely covered with copper film, and the microstrip wire is short-circuited to the ground by electroplating copper on the side, then connect the probe to the plug-type SMA connector. The characteristic impedance of the transmission line is matched to approximately 50 Ω. The film is placed on a heater with one side up, the sample position is controlled through a position control system. The film is placed below the microstrip line. In order to ensure the high accuracy of measurement results, the air gap is required to be less than 0.5 mm. The schematic diagram of the whole test system is shown in Fig. 17(c). The four-part method is used to calculate the thin film magnetic spectrum.[11]

In this study, the permeability values of CoZr films at different temperatures are studied as shown in Fig. 18. With temperature increasing from 25 °C to 200 °C, the peak value of magnetic conductivity shifts towards the low frequency, while the value of the real part of magnetic conductivity increases gradually with the increase of temperature in the low frequency band and at the resonance frequency.

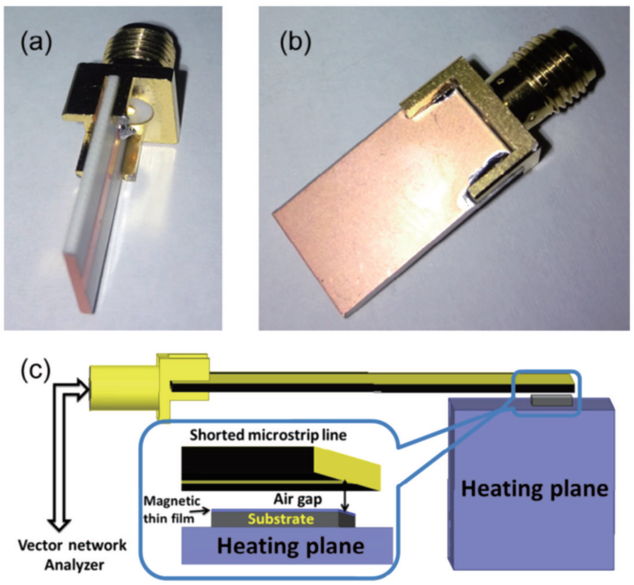


Fig. 17. (a) Side and (b) back view of the designed shorted microstrip line probe, (c) schematic diagram of high temperature magnetic spectrum testing device.[81]

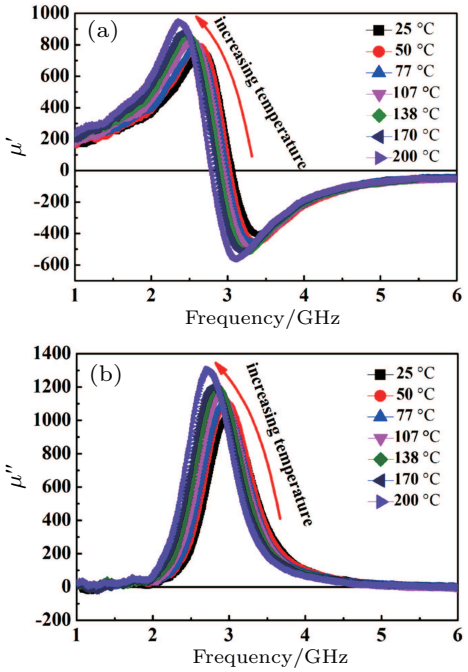


Fig. 18. (a) Real and (b) imaginary parts of permeability spectra of CoZr films at different temperature points.[81]

The resonance frequency, by fitting all the spectra with the theoretical equation, decreases from 3.01 GHz to 2.71 GHz when the temperature increases from 25 °C to 200 °C as shown in Fig. 19. This experimental phenomenon is consistent with the Kittel equation for calculating thin film FMR,

which is similar to the change trend of uniaxial anisotropy H_k with T.[82] The dynamic susceptibility can be obtained from the magnetic spectra. The static susceptibility can be calculated with the saturated magnetization and the static anisotropy field. As shown in Fig. 19, the static result is in good agreement with the dynamic one.

Due to the non-contact and simple design of the probe, this method has many advantages. First, the measuring temperature range of this method can be extended up to 200 °C. If the normal PCB is replaced with an alumina PCB, the test temperature can also be raised to a higher value. Second, the probe is expected to be used in other test methods. For example, it is used to study spin rectification effect and microwave assisted magnetization reversal.[83–85]

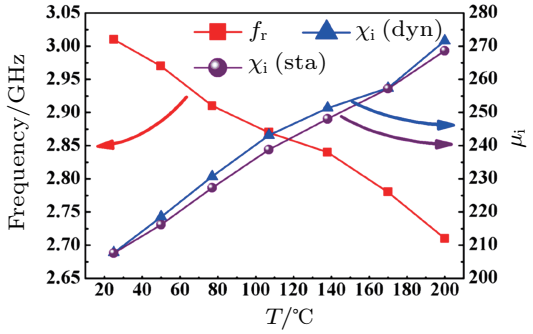


Fig. 19. Plots of resonance frequency f_r and initial permeability μ_i versus temperature of CoZr films.[28]

8. Summary

The basic principles and measurement details behind the design of various microwave permeability testing devices have been introduced. Each method has its own advantages and disadvantages, so an appropriate test method can be chosen according to the measurement purposes. Obviously, it is necessary to further study the magnetic permeability characterization methods in higher temperature and frequency range to meet the needs of industrial development. Microscale permeability is another research topic, which may be useful for studying magnetic resonance or microwave dynamics on a nanoscale by using microwaves.

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