Development of Gyromagnetic and Dielectric Materials and Module for Microwave Application

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For microwave components in a communication system, the toroid module is composed of gyromagnetic yttrium iron garnet ($Y_3Fe_5O_{12}$ or YIG) elements and TiO_2 ($\varepsilon \approx 100$) dielectric elements; the transformer module is composed of Mg_2SiO_4 ($\varepsilon \approx 6$) and $BaTi_4O_9$ ($\varepsilon \approx 38$) dielectric elements. This research starts from the perspective of establishing measurement instruments and analysis methods to overcome the bottleneck of high-frequency microwave characteristics analysis, and then investigate the optimized formulations and process conditions of the gyromagnetic and dielectric materials for the microwave characteristics of the desired frequency band. According to the selected operational frequency band, the appropriate Mn-Al substituted YIG quarter nary system is designed for gyromagnetic elements use. Besides, the three dielectric materials TiO_2, Mg_2SiO_4 and $BaTi_4O_9$ with suitable additives are determined to meet the requirements for toroid module and transformer module, respectively. Subsequently, all materials and processes are integrated to develop the toroid and transformer modules and assemble into required microwave components. Finally, in accordance with user needs, module production and component assembly mass production technologies have been set up to meet the strict requirement of magnetic properties and microwave component characteristics: (1) saturation magnetization ($4\pi M_s$) = 690±20 Gauss; (2) the squareness of hysteresis loop curve > 0.89; (3) the ferromagnetic resonance linewidth (ΔH)≤ 30; (4) the spin-wave resonance linewidth (ΔH_k)≥ 4; (5) the insertion loss (S_{21}) < 0.9dB; (5) the reflection loss (S_{11})>16dB; (7) the phase shift deviation at different testing temperature (@23°C, 43°C, 64°C) < 5°. With the public's attention, the 5G system would be fully commercialized in 2020, which will bring the commercial application of this microwave module developed by this study.

Keywords: Gyromagnetic; Dielectric; Microwave; Toroid Module; Transformer Module

1. INTRODUCTION

Magnetic oxide materials have been known for centuries for their magnetic properties as used by mariners in the Middle Ages as a compass is actually magnetite or Fe_3O_4 with the spinel structure. Magnetic oxide materials have the advantage of being insulating so that there are no eddy current losses due to free electrons as in metals. While metals show ferromagnetic behavior due to the electron spin effects caused by the direct interaction between neighboring metal atoms, insulating oxides show ferrimagnetic interactions in which the non-magnetic oxygen ion mediates the magnetic interaction between the second nearest neighbor cations. As a result, the magnetic properties of most oxides are diluted relative to metals. Metal magnets show much higher Curie Temperatures; the temperature at which thermal energy breaks down the magnetic interaction. There are three major structural families of magnetic oxide materials of technological importance: the garnets, spinels, hexagonal ferrites. Hexagonal ferrites show a wide range of useful applications including permanent magnets and absorbers but, because of their high anisotropy, have not been widely used for microwave magnetic applications. Spinels were the earliest ferrite based materials studied and are very useful for a wide range of applications from the kHz range to the 10GHz range. However, most of the activity for microwave magnetic materials centers on the garnet structure and YIG based materials are also called gyromagnetic material for their special properties in the microwave domain. The gyromagnetic material and the manganese zinc and nickel zinc magnetic materials are all oxide soft magnetic materials. The magnetic properties of the manganese zinc and nickel zinc magnetic materials come from the magnetic domain conversion. The speed of the magnetic domain conversion directly affects the operable frequency (refer to Fig.1). On the other hand, the characteristics of the gyromagnetic material come from the electron spin under the static magnetic field, the conversion is fast and the high-frequency loss is small, so it can be used in high-frequency microwave and millimeter-wave communication. The characteristic difference between the gyromagnetic material and the manganese zinc and nickel zinc magnetic materials is shown in Table 1 below [1-2].

Gyromagnetic materials are magnetic materials with a unique microwave characteristic-"Magneto-Optical Effect", which refers to various phenomena encountered when electromagnetic waves propagate in a gyromagnetic material to which a static magnetic field is applied[1-2]. In these gyromagnetic materials, the "left-handed" and "right-handed" and "elliptically polarized waves" can propagate through the medium at different rates, leading to some very important effects, such as the tensor characteristics of magnetic permeability, phase shift, Faraday rotation, resonance absorption, field shift, birefringence, spin wave, etc. The microwave components designed with the above gyromagnetic characteristics are mainly used for the transmission and conversion of microwave energy. Commonly used phase shifters, isolators, circulators, filters, oscillators, attenuators, modulators, etc. applied in military or commercial fields such as radar, communication base station, artificial satellite, etc. The manufacturing and application design techniques of gyromagnetic materials are difficult, and they are among the highest-ranking magnetic materials. They mainly include yttrium iron garnet ($Y_3Fe_5O_{12}$ or YIG series), spinel (AFe_2O_4 spinel series), and hexaferrites ($AFe_{12}O_{19}$ hexagonal crystal system). The difference mainly lies in the application frequency domain of

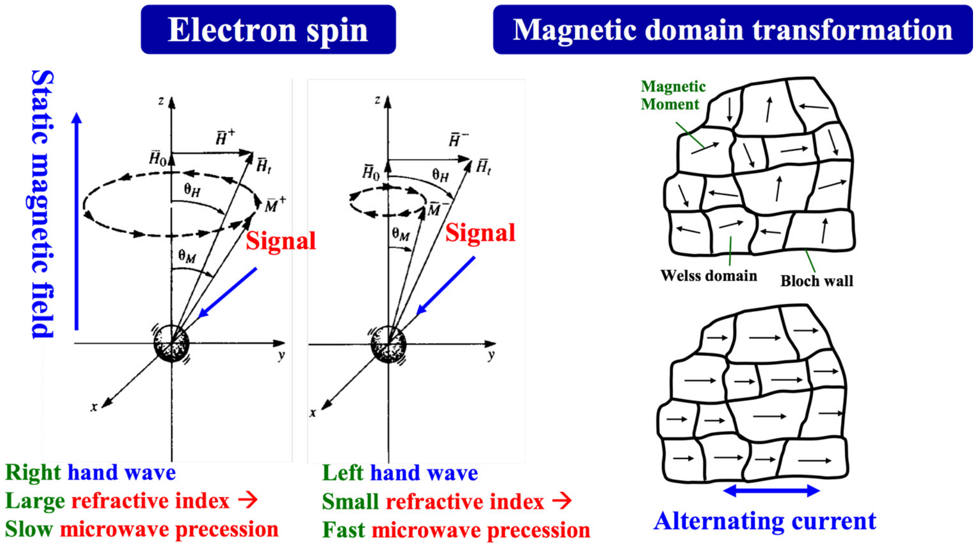


Fig.1. Schematic diagram of the application principle of gyromagnetic and manganese zinc and nickel zinc magnetic materials.

Table 1 The difference between the characteristics of gyromagnetic and manganese zinc materials.

Material	Gyromagnetic material	Mn-Zn magnetic material
Classification	Soft magnetic material	Soft magnetic material
Use environment	Under static magnetic field	Under alternating current (coil winding)
Application Characteristics	Electron spin (Signal or circuit to return line is different	Magnetic domain transformation (Same signal or circuit loop to return line)
Operable frequency	High frequency (0.3GHz~100GHz)	Low frequency (10KHz~1MHz)
Resistivity	$>10^{14}$ Ω-cm	1~10^7 Ω-cm
Loss	Minimal high frequency loss	High frequency loss
Component application	Circulator, phase shifter, isolator, filter And other microwave components	Transformer, inductor
Terminal application	Military radar, mobile base station, Small satellite ground station	Power Supplier

magnetic materials (refer to Fig.2), which is an indispensable material in microwave-millimeter wave (1-50GHz) electronic communication equipment and systems[3-6]. Hexagonal ferrites show a wide range of useful applications including permanent magnets and absorbers but, because of their high anisotropy, have not been widely used for microwave magnetic applications. Spinels were the earliest ferrite based materials studied and are very useful for a wide range of applications from the kHz range to the 10 s of GHz. However, most of the activity for microwave magnetic materials centers on the garnet structure and YIG based materials.

On the other hand, dielectric materials have always been key materials in microwave applications. There are three critical material parameters that need to be considered in the design of a microwave dielectric material. The first, the dielectric constant (ε), can be derived by summing the ionic polarizabilities of the individual ionic components and dividing by the molar volume. The second parameter is the quality factor, or Q. The Q of the material is defined as the inverse of the loss tangent (tanδ). The tanδ is a measure of the response of a material to an applied frequency. In microwave applications, the cavity size as well as the intrinsic tanδ will affect the Q of the material in a cavity resonator. The Q of the material is measured by looking at the bandwidth 3dB below the peak of the primary resonant mode. A higher Q material will have a sharper resonance with an extremely narrow bandwidth leading to sharp cutoffs in filters and the ability to stuff many more channels into a single auto-tuned combiner. A crude rule of thumb for materials is that the product of the Q and the resonant frequency (i.e., Qf product) is a constant. This means that the higher the frequency, the lower the Q value. While the Q does decrease with frequency, the "Qf product rule" is rarely obeyed and the Qf product tends to increase with frequency. The third factor, the τ_f, describes the temperature behavior of the resonator, defined as the temperature coefficient of resonant frequency, measured in parts per million per degrees C. The τ_f is a material property intimately associated with chemical and crystallographic parameters. Designers of microwave elements often require the ceramic component to have a τ_f (or temperature drift) to balance out the intrinsic thermal drift in the metal cavity. That is why in developing dielectric systems, it is important to offer the customer a very large range of available τ_f options. Among the earliest microwave dielectric materials are magnesium titanate ($MgTiO_3$), calcium titanate ($CaTiO_3$), barium tetratitanate ($BaTi_4O_9$) and barium nonatitanate ($Ba_2Ti_9O_{20}$), rutile (TiO_2) and $ZrTiO_4$ type materials. According to the frequency matching design of microwave components, the suitable dielectric materials were determined to meet the specification of microwave components at a given frequency. Taking the consideration for dielectric materials, the Q value and Qf product are usually used to evaluate the performance of dielectric materials. For the application requirements of dielectric components in microwave domain, the target of the dielectric constant is "Nominal The Best", and a little deviation is not allowed, otherwise, these slight deviations will have a considerable impact on the characteristics of the overall phase shifter, which is why the vast majority of companies in the world cannot produce dielectric materials for microwaves.

Aiming at the gyromagnetic material, dielectric

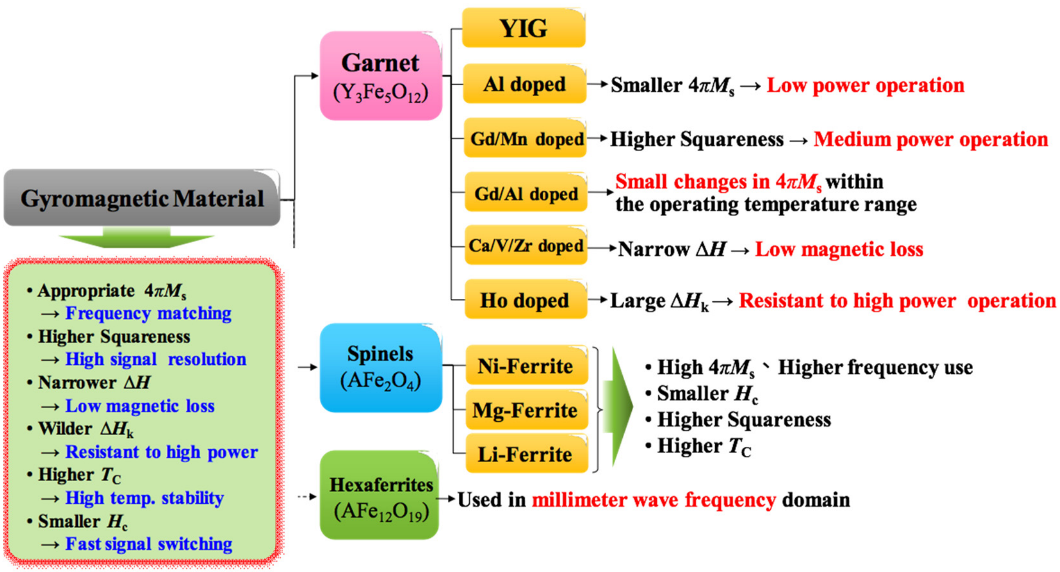


Fig.2. Gyromagnetic material classification.

material and their application development, the research is carried out on the microwave theory, the analysis of microwave characteristics of materials, the establishment of measurement instruments, the development of various key material formulations and process technology and the establishment of component module manufacturing technology in order to set up the relevant technology of key microwave modules.

2. EXPERIMENTAL METHOD

2.1 Establishment of gyromagnetic property measurement technology and equipment

The index parameters of the gyromagnetic material and components and the corresponding measurement equipment are shown in Table 2. The magnetic properties and material properties, such as saturated magnetic flux density (B_s), residual magnetization (B_r), coercive force (H_c), angle ratio of hysteresis curve (Squareness), saturation magnetization ($4\pi M_s$), dielectric coefficient (ε), and dielectric loss ($\tan\delta$) are the "Key threshold" test criteria for judging whether gyromagnetic materials are suitable. Based on our past development in the field of magnetic materials, the fast and stable detection method of characteristics and magnetic properties of B_s, B_r, H_c, Squareness, $4\pi M_s$, ε, $\tan\delta$, etc. for gyromagnetic materials in the DC (applied magnetic field is zero) state are first established in this work.

In addition, the ferromagnetic resonance linewidth (ΔH) is an important parameter to measure the high-frequency magnetic loss of the gyromagnetic material, and the spin wave resonance line needs to be considered when studying the relaxation characteristics of the gyromagnetic material and the analysis of spin wave resonance line width (ΔH_k) is required for the change of characteristics applied to high-power components. The detection principle of $4\pi M_s$, ε, $\tan\delta$, and ΔH is shown in Fig.3. For the detection of $4\pi M_s$, it is necessary to prepare a 19.05mm diameter and 2.54mm thick sheet-shaped gyromagnetic piece for detection with a Gauss meter. For ε and tan δ, it is necessary to prepare a gyromagnetic rod with a length of >12mm and a diameter of 1mm and place it in a special waveguide resonance cavity for detection by a network analyzer. For ΔH, the preparation of a diameter of 1.5mm, and a spherical gyromagnetic sample with no surface cracks on the polished surface are required, and it is detected by a network analyzer in the special waveguide resonance cavity. At present, we have established a fast and stable detection method for the magnetic properties and material properties of B_s, B_r, H_c, angular ratio, $4\pi M_s$, ε, $\tan\delta$, ΔH, etc. of the gyromagnetic materials in the direct current (DC) state, but ΔH_{eff} and other important microwave

Table 2 Gyromagnetic material and component characteristic testing equipment

Characteristic	Physical Quantity		Analysis Equipment
DC Magnetic Characteristics	Saturated magnetic flux density	B_s (G)	B-H Loop Tracer
	Residual magnetization	B_r (G)	
	Coercive force	H_c (Oe)	
	Squareness ratio of hysteresis curve	Squareness	
Microwave Characteristics	Saturation magnetization in microwave frequency band	$4\pi M_s$	The new ferromagnetic resonance instrument (developed by this research)
	Dielectric constant	ε	
	Dielectric loss	$\tan\delta$	
	Ferromagnetic linewidth	ΔH (Oe)	
	Equivalent ferromagnetic resonance linewidth	Δh_{eff} (Oe)	
	Spin wave resonance linewidth	Δh_k (Oe)	
	Curie temperature	T_c (°C)	Impedance Analyzer / Temperature Control Box
	Permeability	μ	VSM / Impedance Analyzer
Phase Shifter Module Features	Return Loss	S_{11} (dB)	Network Analyszer
	Insertion loss	S_{21} (dB)	
	Phase shift deviation	°	

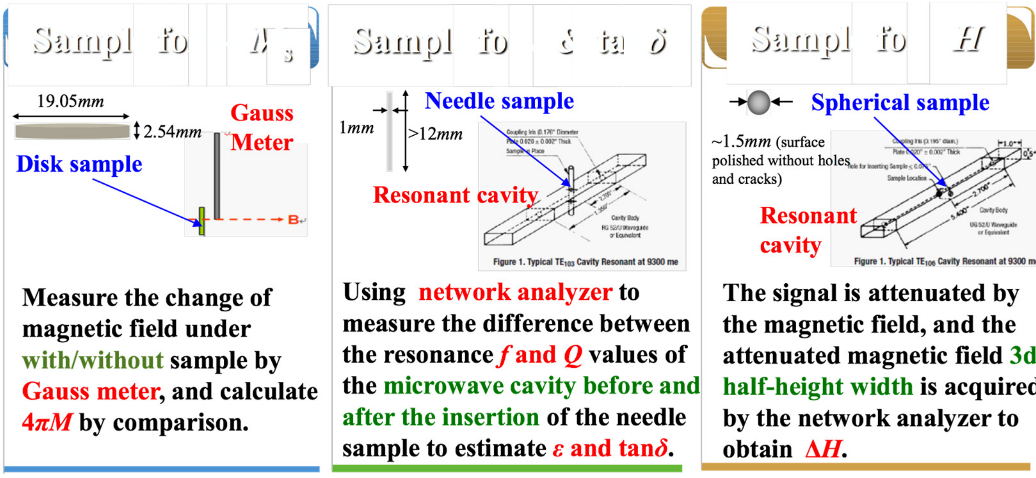


Fig.3. The detection principle of 4πMs, ε, tanδ and ΔH of gyromagnetic materials.

gyromagnetic properties such as ΔH_k cannot be resolved. Besides, the preparation of the required test samples for $4\pi M_s$, ε, tanδ and ΔH is relatively complicated and takes too long time. In order to build a simple, fast and complete microwave gyromagnetic characteristic analysis technology, this research goes further and even related detection technology development and equipment construction.

In this work, based on the ferromagnetic resonance theory, we designed an X-band (8-12GHz) frequency band microwave resonant cavity that can analyze the resonance absorption spectrum of gyromagnetic materials such as Garnet and Spinel. Next, we further analyze the full width at half maximum width (FWHM) of the peak of the resonance spectrum to obtain the ΔH measurement parameter. At the same time, based on the derivation of microwave theory of ΔH_k and the analysis of the characteristics of ΔH_k with high-power microwave excitation, a new ferromagnetic resonance instrument is established that can simultaneously analyze to build $4\pi M_s$, ε, tanδ, ΔH, ΔH_{eff} and ΔH_k.

2.2 Establishment of Correlation between gyromagnetic material and corresponding characteristics

In the early stage of development, after the assembly of the module is completed, the test results of the module characteristics are used to determine whether the gyromagnetic material is suitable. The time required for the test is very long, which makes product development extremely difficult. Our improvement concept is to establish the correspondence between the material properties and the module, so the certification time can be greatly shortened, and the product development and order taking ability can be effectively strengthened. In this study, through software simulation, the correlation between the gyromagnetic material and the characteristics of the microwave component was established, and the corresponding indexes of the gyromagnetic material's size, processing problems, surface characteristics and gyromagnetic material characteristics were established for the microwave component characteristics. In addition, with this result, we also successfully designed and developed microwave commercial circulator components for 900MHz, 1800MHz and higher frequencies domain, which can be applied to commercial base stations.

2.3 Development of microwave gyromagnetic materials

The phase shifter is an important component in the microwave communication system. It changes the phase consistency of the microwave components to improve the synthesis efficiency of the output power of the microwave components or the synthesis efficiency of the echo signals, and improve the monitoring ability of the communication system. The requirements for it are accurate phase shift values (larger squareness of hysteresis curve), stable performance (small ΔH), wide-band operation (applicable B_s, $4\pi M_s$), low loss (small ΔH, tanδ), high power capacity (ΔH_k should be large), easy to control quickly (H_c should be small), etc(1-2). Before the invention of the electronic phase shifter in 1950, almost no matter whether it was a variable phase shifter or an invariable phase shifter, they were all mechanical types, so their practicality was therefore limited, while the appearance of the electronic phase shifter thus represents a new generation of special significance. For example, in 1957, Reggia Spencer made a ferrite phase shifter for radioactive scanning antenna phase array. This phase shifter is an oxide phase shifter made for radioactive scanning antenna phase array. The amount of electromagnetic wave phase shift can be expressed by equation (1). Among them, the magnetic permeability (μ) can be adjusted by changing the strength of the external magnetic field to achieve the purpose of phase shift.

Electromagnetic wave phase shift: (Φ) =

$2\pi f L (\mu\varepsilon)^{1/2}$ .... (1)

(f: frequency, L: path length, μ: magnetic permeability, ε: dielectric constant)

In this study, the design flow chart of in Fig.4 for the gyromagnetic material is developed, aiming at the gyromagnetic material that can meet the above characteristics at the same time.

2.4 Development of microwave dielectric materials

Although the phase shift function mainly comes from the gyromagnetic material, for the toroid module in the phase shifter, the function of the K100 dielectric element is to concentrate the electromagnetic wave energy at the center of the waveguide, because most electromagnetic waves here (as shown in Fig.5), the K100 dielectric element has a huge impact on the overall functional quality of the phase shifter. On the other hand, for transformer module, K6 and K38 dielectric components are just like the buffer of electromagnetic waves in the wave guide tube. The electromagnetic wave signal can be converted before entering the wave guide tube. It is also very important for reducing the overall energy loss of the phase shifter and improving the efficiency. Strictly speaking, for the insertion loss and return loss of the phase shifter module, the electromagnetic wave enters from the dielectric element K6 and passes through the interface (adhesive) of dielectric K6/K38 element, K38, K38/toroid module interface (adhesive) and toroid module (K100/ adhesive/Garnet gyromagnetic material), then go out from the K38/K6 interface at the other end. These gyromagnetic components, dielectric components, and bonded interfaces all affect insertion loss and reflection loss. The smaller the insertion loss, the better, indicates that most of the electromagnetic wave energy can pass through. The smaller the insertion loss, the greater the reflection loss is. For the application requirements of dielectric components in the phase shifter, the target of the dielectric constant is "Nominal at Best", and a little deviation is not allowed, otherwise serious impacts to the overall characteristics of the phase shifter occur, which is why most companies cannot produce dielectric components for microwave components. The conditions and specifications of dielectric components K6, K38, and K100 refer to Table 3.

This research firstly develops the most important K100 dielectric component (in the toroid module) of the phase shifter element, and its development flow chart is the same as above gyromagnetic materials in Fig.4.

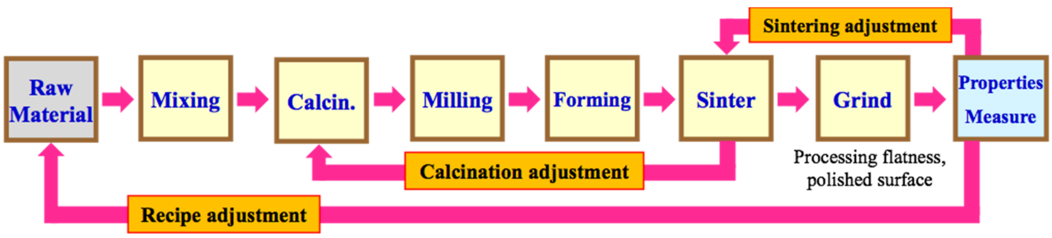


Fig.4. Development flow chart of gyromagnetic materials.

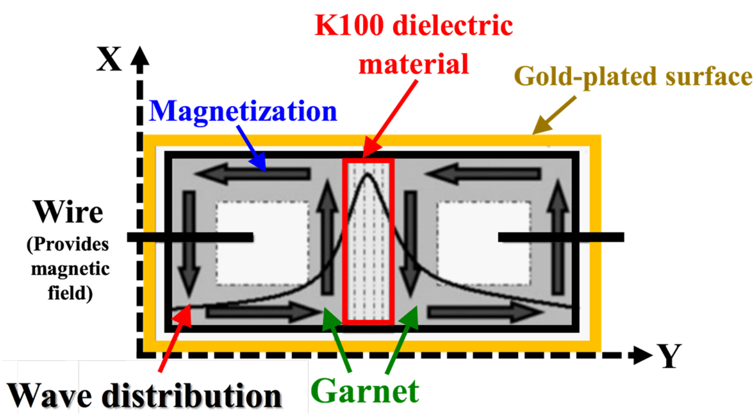


Fig.5. Function of dielectric component of toroid module in phase shifter.

Table 3 Specification of dielectric components in phase shifter element (including Toroid and Transformer modules).

Material	Dielectric Constant (ε)	Dielectric loss (tan δ)	Q×f(GHz)	Material System
K100	99~101	<0.1	>12,000	TiO_2
K38	37~38	<0.05	>20,000	$BaTi_4O_9$
K6	6~6.6	<0.02	>20,000	Mg_2SiO_4

3. RESULTS AND DISCUSSION

3.1 Establishment of gyromagnetic property measurement technology and equipment

In this study, a fast and stable detection method for magnetic characteristics and material characteristics such as saturation magnetic flux density (B_s), residual magnetization (B_r), coercive force (H_c), verticality of hysteresis curve (Squareness), saturation magnetization ($4\pi M_s$), dielectric constant (ε) and dielectric loss (tanδ) of the gyromagnetic material in the DC state were first established. Using this test method as the first level of detection standard, the test results would determine whether the gyromagnetic material is suitable. Nevertheless, because microwave gyromagnetic materials are high-end materials among magnetic materials, in addition to the relatively complicated formulation and manufacturing process, the required magnetic materials are designed for the detection of the magnetic properties, such as ferromagnetic resonance linewidth (ΔH) and spin wave resonance linewidth (ΔH_k) in the microwave frequency band is also the most critical link among them. Early, the research team lacked the ability to analyze and detect ΔH and ΔH_k, and thus, it is impossible to correctly control the characteristics of the magnetic material when it is used in microwave domain and in the high power range. In order to fill the gap in this detection technology, it is further invested in the establishment the detection technology of ΔH and ΔH_k, and has completed the construction and verification of new measurement equipment in 2017. In the research results(7-8), based on the theory of microwave and spin wave resonance, a special microwave resonance cavity was designed, which was matched with high-power microwave excitation, and the half-width of resonance was captured by a network analyzer and complete analysis of microwave gyromagnetic characteristics of microwave gyromagnetic characteristics detection technology and equipment. A new type of ferromagnetic resonance analyzer that can analyze $4\pi M_s$, ε, tanδ, ΔH, ΔH_{eff}, and ΔH_k is built, and the square measurement sample required by this system is easy to prepare. Different from the original measurement of $4\pi M_s$, ε, tanδ, ΔH, which requires the use of samples of special sizes and shapes, it can greatly shorten the measurement time. In addition, it also solves the problem that ΔH, ΔH_{eff}, and ΔH_k cannot be measured in the past. The measurement system and measurement results are shown in Fig.6. By completing the above detection technology and measurement equipment of the characteristics of B_r, H_c, Squareness, $4\pi M_s$, ε, tanδ, ΔH, ΔH_{eff} and ΔH_k of the gyromagnetic material, in addition

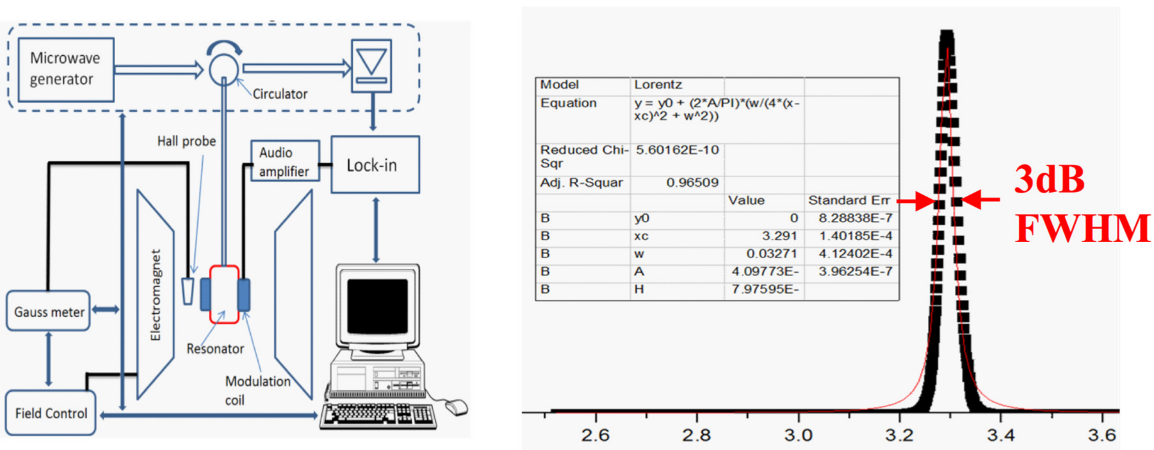


Fig.6. The new type of ferromagnetic resonance instrument established in this study that can simultaneously analyze $4\pi M_s$, ε, tanδ, ΔH, ΔH_{eff} and ΔH_k.

to assisting the evaluation of the formula characteristics of the gyromagnetic material, the detection and analysis results have also become an important basis for quality control of shipments. This research result plays an important role in the subsequent development of microwave gyromagnetic products.

3.2 Establishment of Correlation between gyromagnetic material and corresponding characteristics

At the beginning of the research, we only knew more about the formula of the gyromagnetic material and the process technology of the gyromagnetic material, but the application of the gyromagnetic material and the application principle are less discussed. The characteristics of the microwave elements such as phase shifter, circulator or isolator are mainly lies in the design of the resonant cavity of the waveguide, the characteristics of the conductor material and the gyromagnetic material, etc. Generally, the user does not have testing equipment to evaluate the characteristics of the gyromagnetic material. The quality of the gyromagnetic material needs to be evaluated by the characteristics of the module (i.e., S Parameters: S_{11}, S_{12}, S_{21}, S_{22}) after assembling through the module. Usually, it's impossible to directly determine the solution to improve the gyromagnetic material used internally, and the time for testing and certification is long. Therefore, this makes it extremely difficult to expand the market of gyromagnetic materials and microwave components. In the research, the correlation between gyromagnetic material and corresponding characteristics of the microwave component has been completed, including the size of the gyromagnetic material, processing problems, surface characteristics and the impacts of the gyromagnetic material characteristics on the properties of the microwave module(7-8). Furthermore, because the correlation is established, it is easier to communicate with the terminal module user later to clarify the gyromagnetic material problem or the module assembly design problem, which can greatly shorten the certification time and effectively strengthen product development and order taking capabilities. It can be seen from the results in the Table 4 below, taking the B_s of gyromagnetic material as an example; whether the microwave parameters S_{21} (Insertion loss) and S_{11} (Reflection loss) of the module meet the requirements can be determined by measuring the magnetic properties B_s of gyromagnetic material.

3.3 Development of microwave gyromagnetic materials

In the research, the following research methods were used: (1) According to the frequency matching design of microwave components, the gyromagnetic material system was determined, the main raw material Fe_2O_3 was selected, and the appropriate physical and chemical iron oxide was chosen as the main raw materials. Then, by adjusting the main components of Fe and Y together with specially modulated trace additives of Mn and Al, establish a Garnet-based gyromagnetic material formulation database for phase shifters. Finally, by comparing with the magnetic properties of commercial components, the most appropriate $Y_3Mn_xAl_yFe_{5-x-y}O_{12}$ iron-poor formula quaternary system was designed for phase shifter use, so that the important magnetic characteristics, such as saturation magnetic flux density reaches the level of commercial products (B_s=560±50Gauss), as well as residual magnetism (B_r), coercive force (H_c), Squareness (>0.89), saturation magnetization ($4\pi M_s$= 690±20G), dielectric constant (ε=14±0.7), dielectric loss

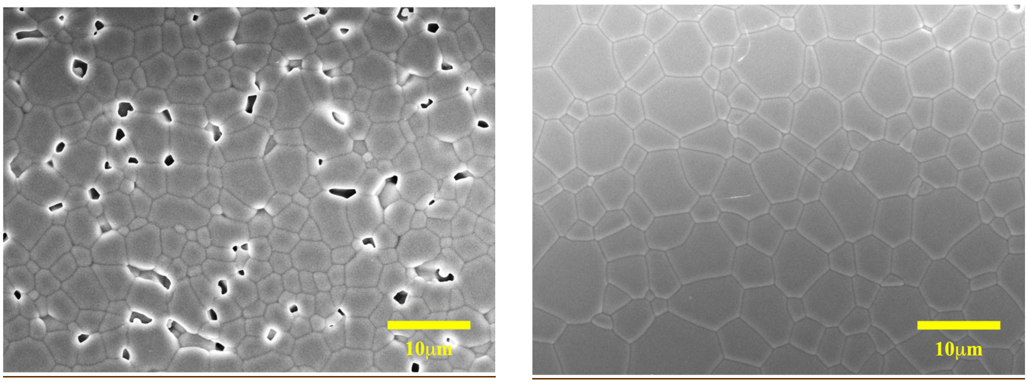
Table 4 Correlation between the properties of gyromagnetic materials and components.

Parameters of Gyromagnetic Materials	Center Frequency	S21	S11	Phase Shift Deviation	High Power Endurance	Component Speed
ε ↑	↓	↓	-	-	-	-
tanδ ↑	-	↑	↓	-	-	-
$4\pi M_s$ ↑	↓	↑	↓	-	-	-
ΔH ↓	-	↓	↑	-	-	-
ΔH_k ↑	-	-	-	-	↑	-
B_s value of hysteresis curve ↑	↓	↑	↓	-	-	-
H_c value of hysteresis curve ↓	-	↓	-	↓	-	↑
Squareness of hysteresis curve ↑	-	↓	-	↓	-	↑
Flatness of surface processing of Gyromagnetic Materials ↑	-	↓	↓	-	-	-

($\tan\delta << 2.5\times10^{-4}$), ferromagnetic resonance linewidth ($\Delta H \leq 30$) and spin-wave resonance linewidth ($\Delta H_k \geq 4$) meet the requirements of the customer. (2) The high oxygen partial pressure sintering process was adopted to increase the impedance value so that the product is suitable for high frequency use (3.2GHz), and the sintering temperature was adjusted to further reduce the coercive force (H_c) to <1Oe, to avoid excessive magnetic loss. Finally, a suitable formula was designated and transferred to manufacture toroid modules. As can be seen from the microstructure of the gyromagnetic material in Fig.7, after these improvements, the phenomenon of uneven grain size and excessive pores encountered in the initial stage of the development of the gyromagnetic material has been overcome, and the overall gyromagnetic characteristics have been significantly upgraded (9-13).

3.4 Development of microwave dielectric materials

In this study, we first analyzed the K100 dielectric component based on the operable frequency of phase shifter and found out that it must use the rutile (TiO_2) material system, and then learned from the theory that the closer the dielectric constant is to 100 and the lower the dielectric loss is better. In the early stage of development, in the high temperature sintering process of TiO_2, abnormal grain growth is easy to occur, resulting in high dielectric constant and dielectric loss, which will affect the characteristics of the phase shifter module. The reason is that TiO_2 is easy to produce reduction phenomenon ($Ti^{4+} \rightarrow Ti^{3+}$) during high temperature sintering. Originally $Ti^{4+}O_2$, but after reduction to $Ti_x^{3+}Ti_{1-x}^{4+}O_{2-x}$, due to the formation of oxygen vacancies, the surface energy of the (101) crystal plane is low and it is easy to grow abnormally. In this study, the introduction of pentavalent additives to achieve the cation valence compensation, but the ion radius must be considered to select more suitable additives for ion replacement. With the addition of trace additives, the sintering temperature can also be reduced, and at the same time the sintering density can reach a densification result greater than 98% of the theoretical density. By designing this specially formulated trace additive formula, it can be seen from the XRD analysis results in Fig.8 that the abnormal growth



(a) (b)

Fig.7. SEM analysis results of (a) before improvement and (b) after improvement of the gyromagnetic material. After adjusting additives and process conditions, the phenomenon of excessive pores and uneven grain size has been improved.

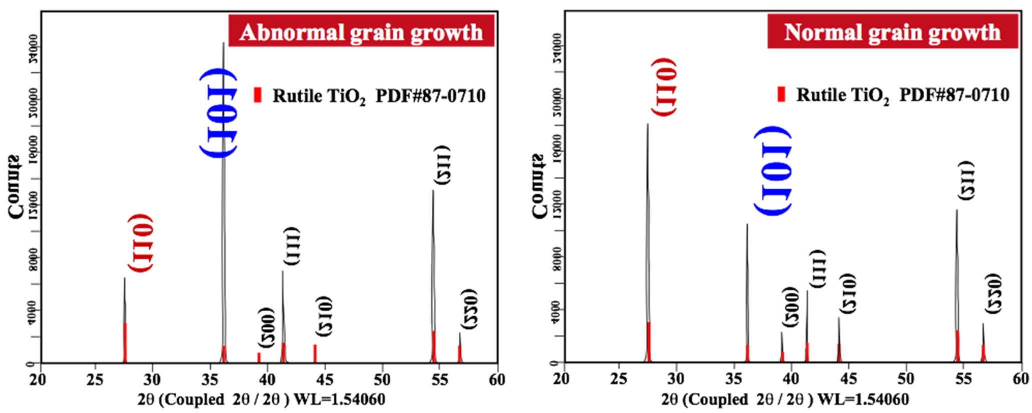


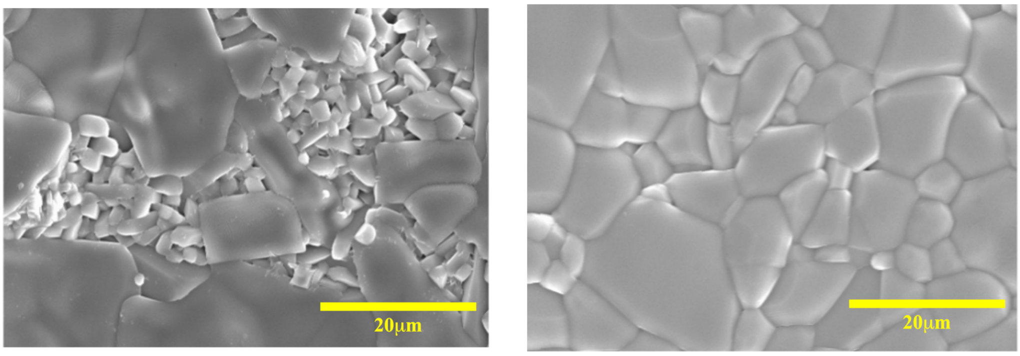
Fig.8. XRD analysis results of K100 dielectric components before and after material formulation optimization.

of the (101) crystal plane is significantly reduced, successfully overcoming the abnormal grain growth of TiO_2. It can also be seen from Fig.9 that after the optimization of the dielectric material, the overall microstructure is dense and without abnormal grain growth. In addition to K100 dielectric materials, including K6, K38, and these three dielectric materials are characterized by the change rate of dielectric constant ε with temperature less than 4ppm, so that the material characteristics could tend to be quite stable. The SEM results of the K6 and K38 dielectric materials after sintering refer to Fig.10 and are dense and free of holes after sintering. After the K6 and K38 dielectric components are integrated into the transformer module and then assembled with the toroid module, the overall characteristics of phase shifter could successfully meet the specifications of the customer.

3.5 Establishment of Manufacturing Process of Phase Shifter Toroid module

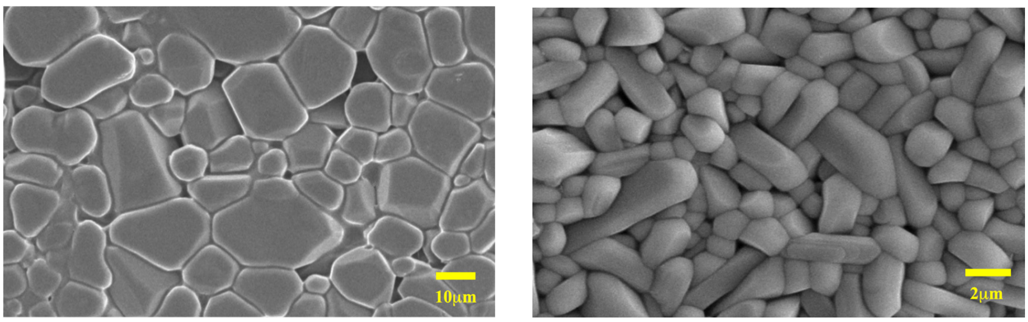
During the integration of the phase shifter, we found that by improving the processing accuracy of the gyromagnetic material and the dielectric material, the establishment of adhesive assembly of the toroid module and the transformer module and the surface gold coating technology can all have improved characteristics for the phase shifter. However, only through these general optimization procedures, the S_{21} of the module is still too high, resulting in problems such as customer returns. After many experiments to explore the reason, it is caused by the difference in the surface processing of the toroid module after the gyromagnetic material is processed and assembled into the toroid module.

In order to solve the problem of high S_{21} of the module, it is improved by increasing the grinding accuracy of the gyromagnetic material, establishing the bonding assembly of the module, and the surface gold coating technology. In the processing of magnetic block using in the toroid module, the processing accuracy must be controlled within ±0.01mm, and the verticality and flatness of the surface must be controlled within <0.005mm, and the bonding area must be polished and the polishing roughness needs to be controlled at <0.2μm. In addition to the above requirements, due to the thickness of the magnetic material being too thin (<1.5mm), special attention needs to be paid to the bending status after processing. On the other hand, because the size is small, and the gyromagnetic material is a soft magnetic material, it



(a) (b)

Fig.9. **S**EM analysis results of K100 dielectric component materials (a) before improvement and (b) after improvement. The formula has been adjusted with additives and process conditions to improve abnormal grain growth.



(a) (b)

Fig.10. SEM analysis results of (a) K6 and (b) K38 dielectric components.

cannot be directly adsorbed on the processing platform by using magnetism, so the grinding process of the grinding wheel needs to consider the number of diamond grinding wheels to polish. The cement used in the polishing process is selected to be softer in accordance with the wear condition of the diamond layer; the processing speed and the feed speed need to be changed in accordance with the number of grinding wheels. Above-mentioned cutting, polishing and grinding parallelism, roughness, and the processing and assembly accuracy of the gyromagnetic materials must be strictly controlled; otherwise the air gap will occur after one magnetic block bonding and assembly with the other which will be serious affect module characteristics. In addition, on the surface gold coating technology of toroid module, the function of surface gold coating is to help the waveguide of the phase shifter module to have the effect of grounding and shielding. The surface gold coating process is first to clean the surface of the sample, pickle with oxalic acid, and then pick Argon plasma as the surface treatment. Immediately, the sputtered TiW alloy layer and the sputtered Cu layer are sequentially deposited; then the Cu layer is plated, and finally the Au layer is plated. The TiW and Cu layers are used as seeding layer to increase adhesion.

At the beginning of the development of phase shifter, we encountered problems in that the toroid module had a low squareness and S_{21} was high (>1dB). In this study, it was found that generally high S_{21} is more likely to occur in a lower magnetic field state. The reason for this low magnetic field loss is that if the external magnetic field strength of the module is not enough to make the magnetic domains in the gyromagnetic material inside the module reach full saturation or complete alignment, it is easier to produce low field loss. Therefore, by moderately reducing the DC magnetic property B_s value of the gyromagnetic material in this study, it can ensure that the gyromagnetic material is fully saturated at a low applied magnetic field strength, so it is less likely to produce low field loss, so S_{21} decreases, which is also a key technology that cannot be obtained from abroad[12-15]. This research further introduces the patented technology that can predict the characteristics of the module in section 3.2 of this article.

Above measurement technology of microwave, detection technology of material property, prediction technology of module property, material formulation and production method of Garnet gyromagnetic material and K100, K38, K6 dielectric material, assembly and mass production technology of phase shifter and other core technologies have been implemented in production. After the integration of toroid modules and transformer modules and combined into phase shifter, its insertion loss S_{21} <1dB, reflection loss S_{11} is more than 16dB, and the phase shift deviation at different testing temperature (@23°C, 43°C, 64°C) is less than 5°, which have successfully met the specifications of the component.

4. CONCLUSIONS

In this study, firstly, based on the ferromagnetic resonance theory, the new type ferromagnetic resonance instrument and the material characteristic analysis method were established to provide us a powerful tool to evaluate the important characteristics of material, such as $4\pi M_s$, ΔH and ΔH_k. And then used the innovative detection technology as the foundation, the Mn-Al substituted garnet gyromagnetic materials ($Y_3Mn_xAl_yFe_{5-x-y}O_{12}$) and dielectric materials (TiO_2, Mg_2SiO_4, $BaTi_4O_9$) with optimized additives are designed to meet low-loss needs and the specification for the desired frequency band at the same time. Furthermore, the cutting, grinding, polishing and adhesion processes are all built up for assembly of gyromagnetic and dielectric elements. Finally, the toroid module integrated with transformer module are successfully established in mass production to meet the strict requirement of magnetic properties and microwave component characteristics: (1) saturation magnetization ($4\pi M_s$) = 690±20 Gauss; (2) the squareness of hysteresis loop curve > 0.89; (3) the ferromagnetic resonance linewidth (ΔH)≤ 30; (4) the spin-wave resonance linewidth (ΔH_k) ≥ 4; (5) the insertion loss (S_{21}) < 0.9dB; (5) the reflection loss (S_{11})>16dB; (7) the phase shift deviation at different testing temperature (@23°C, 43°C, 64°C)< 5°. Based on above result, the microwave component realized in this study could satisfy the different demands in the operational frequency band and also have related application performance like radar system. In the near future, the frequency-selective materials and elements developed in this wok will continue to be required for commercial fields such as 5G base stations and automobile collision avoidance systems in everyday life.

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REFERENCES

1. V. G. Harris, A. Geiler, Y. J. Chen, Recent advances in processing and applications of microwave ferrites, *J. Magn. Magn. Mater*, 2009, 321:2035-2047.
2. G. P. Ridrigue, Magnetism in Microwave Devices, *J. Appl. Phys*., 1969, 40:929-938.
3. J. Čermák, R. Novák, P. Novák, M. Nevǎiva, Yttrium Iron Garnet Films Substituted by Gd and. Mn, *Physica Status Solidi (a)*, 1984, 85, 173-177.

4. M. A. Gilleo, S. Geller, Magnetic and Crystallographic Properties of Substituted Yttrium-Iron Garnet, $3Y_2O_3{\cdot}xM_2O_3{\cdot}(5\text{-}x)Fe_2O_3$, *Phys. Rev.*, 1958, 110: 73-78.
5. D. H. Temme, Progress and problems in high power phasers for array radar, *IEEE NEREM Rec.*, 1964, 6, 138-140.
6. J. Douglas Adam, Lionel E. Davis, Gerald F. Dionne, Ernst F. Schloemann and Steven N. Stitzer, Ferrite Devices and Materials , *IEEE Trans. Microwave Theory Tech.*, 2002, vol. 50, 721-736.
7. U. Hoeppe, Smart measurement setup for spin-wave linewidth ΔH_k, *IEEE Transactions on Magnetics*, Sep. 2002, vol.38, no.5, 3126-3128.
8. Nan Mo, Jerome J. Green, Bailey A. Beitscher and Carl E. Patton, High precision metrology based microwave effective linewidth measurement technique, *Review of Scientific Instruments.*, 2007, 78, 113903.
9. Ching-Chien Huang, Chih-Chieh Mo, Yung-Hsiung Hung, Wei-Zong Zuo, Jing-Yi Huang, Hsiao-Hsuan Hsu, and Chun-Hu Cheng, Effect of Particle Size of as-milled powders on Microstructural and Magnetic Properties of $Y_3Mn_xAl_{0.8\text{-}x}Fe_{4.2}O_{12}$ Ferrites, *Journal of the American Ceramic Society*, 2018, 10.1111/jace.16211, 1-10.
10. Ching-Chien Huang, Wei-Zong Zuo, Yung-Hsiung Hung, Jing-Yi Huang, Ming-Feng Kuo and Chun-Hu Cheng, Effect of Specific Surface Area of Raw Material Fe_2O_3 on Magnetic Properties of YIG, *Journal of Magnetism and Magnetic Materials*, 2018, vol. 449, 157-164.
11. Ching-Chien Huang, Yung-Hsiung Hung, Jing-Yi Huang and Ming-Feng Kuo, Performance Improvement of S-Band Phase Shifter Using Al, Mn and Gd doped $Y_3Fe_5O_{12}$ and Sintering Optimization, *Journal of Alloys and Compound*, 2015, 643, S193-S198.
12. Ching-Chien Huang, Yung-Hsiung Hung, Jing-Yi Huang and Ming-Feng Kuo, “Impact of Stoichiometry and Sintering Temperature to Magnetic Properties of $Y_3Mn_xAl_{0.83\text{-}x}Fe_{4.17}O_{12}$ Ferrites, *IEEE Transactions on Magnetics*, 2014, vol. 50, no. 8, 2801004.
13. Ming-Feng Kuo, Yung-Hsiung Hung, Jing-Yi Huang, and Ching-Chien Huang, “Substitution effects on magnetic properties of $Mg_{1.3\text{-}x}Mn_xAl_yFe_{1.8\text{-}y}O_4$ ferrite”, *AIP Advances*, 2017, vol. 7, 056104.
14. H. Kurebayashi, O. Dzyapko, V. E. Demidov, D. Fang, A. J. Ferguson, and S. O. Demokritov, Spin pumping by parametrically excited short-wavelength spin waves, *Applied Physics Letters.*, 2011, 99, 162502.
15. Gerald F. Dionne, Patricia J. Paul, and Russell G. West, Compensation of Magnetostriction Effects in Iron Garnets by Manganese Additions, *Journal of Applied Physics*, 1970, 41, 1411. □