AMPEX

ESD-TR-71-267

DEVELOPMENT OF LITHIUM FERRITES FOR ARRAY RADAR PHASERS

Prepared by

Ampex Corporation
Research and Advanced Technology Division
401 Broadway
Redwood City, California 94063

for

Massachusetts Institute of Technology
Lincoln Laboratory

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Under

Purchase Order No. B-130
Prime Contract No. F19628-70-C-0230
ABMDA 7x263304D215

This Report Covers the Period

1 July 1969 to 31 August 1971

Ampex Report No. RR 71-13

30 August 1971

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ABSTRACT

Lithium ferrites are low cost materials which are attractive for microwave device applications. For a number of years these materials have dominated the computer core industry because of excellent temperature performance and squareness of hysteresis loops. Previous attempts at exploiting lithium ferrites for microwave applications have met with limited success. Properties which required further improvements were the dielectric and magnetic loss tangents, anisotropy, coercive force, and density. This report discusses the development of microwave S- and C-band lithium ferrites with substantially improved properties, comparable to those of the more expensive ferrimagnetic garnets currently in use. Although the development of these materials has been directed at applications for latching microwave devices, they are also useful for other microwave devices.

The requirements for ferrite materials in latching phasers are reviewed, the compositional factors which result in useful properties are discussed, and performance in circulators and latching phasers is reported.

Accepted for the Air Force
Joseph R. Waterman, Lt. Col., USAF
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ACKNOWLEDGEMENT

This program was sponsored by the Department of the Army.

The authors gratefully acknowledge the work of H.B. Im and S. K. Banerjee in the early stages of the program, and extremely helpful technical discussions with Prof. J. Smit. The work of C. Shell, M. Chan, J. Mackey, S. Lichter, and L. Moultrie in the preparation and evaluation of samples, and the technical editing by I. Strassner are also deeply appreciated.

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1.0 INTRODUCTION

1.1 Background

Early in the development of microwave devices, the magnetic materials which were in predominant use were from the magnesium manganese (Mg Mn) and nickel (Ni) ferrite systems. Through the substitution of nonmagnetic ions such as zinc (Zn), aluminum (Al), and gallium (Ga) into these systems, a wide range of saturation magnetizations (ca. 500 to 5000 gauss) had been provided to cover a large segment of the microwave frequency spectrum.

Both the Ni and Mg Mn ferrite systems are characterized by high resistivity ($>10^8$ ohm-cm), good dielectric loss tangents (0.00025 to 0.001), moderate anistoropy fields, and moderate-to-broad resonance linewidths (100 to 800 Oe). The behavior of these two systems diverges with respect to the temperature behavior and the hysteresis loop properties. The Mg Mn system, which has long been is use in computers, has excellent square-loop properties, and has found application in latching microwave devices such as switches, phase shifters, etc. However, the low Curie temperatures in this system result in excessive temperature variations in the saturation magnetization, remanence and coercive force. On the other hand, the Ni system provides adequate temperature performance, but the compositions with acceptable magnetic loss have had poor hysteresis loop properties, precluding their use in latching devices.

With the discovery of the magnetic garnets, the Mg Mn and Ni ferrite systems have been increasingly displaced in a variety of microwave applications. The garnets have provided excellent dielectric and magnetic loss tangents, high densities, low anisotropy, and consequently, a low resonance linewidth. (In dense materials with minimal levels of relaxing ions, the resonance linewidth is predominantly attributable to the anisotropy field.) Unfortunately, the Curie temperatures of the garnets are low ($\leq$ 280°C). However, the substitution of rare earth ions such as gadolinium (Gd) for yttrium (Y) provides good temperature compensation with a compromise in the magnetic loss. The squareness and remanence ratios in the garnet family, while not matching the values obtainable in the Mg Mn system are sufficient for utilization in latching devices. Moreover, the garnets have low coercive forces ($<$ 1 Oe). The reason that the squareness and remanence ratios are not higher is attributable to the low anisotropy of the garnets. A necessary condition for good squareness and remanence ratios is the dominance of the anisotropy energy over the magnetostrictive energy (1). Failure to fulfill this condition also causes the garnets to be stress-sensitive (2). The addition of manganese (Mn) decreases the stress-sensitivity (3,4) but does not eliminate it completely. Innovative device designs (5) which minimize mechanical stresses on the ferrite part can be expected to minimize the deleterious effects of magnetostriction. Despite the few deficiencies of the garnets, the desirable properties mentioned previously have caused the garnet materials to dominate in the microwave industry. For more details on microwave materials and their use in devices, see the review by Soohoo (6).

The high cost of garnet raw materials (i.e., yttrium oxide and rare earth oxides) has led to efforts to evolve lower cost systems. One approach has been to replace yttrium with cheaper materials such as calcium (7 - 9). Although this scheme has advantages and the resulting materials will be useful replacements for the more costly garnets in some devices, the success up to this point has been limited, and more exploration is necessary. The two major problems to overcome are remanence ratios which are too low for latching device application and poor temperature performance.

Another approach to finding low cost replacements for garnets has been the development of microwave lithium ferrite compositions, which is the subject of this report. Lithium ferrites have been of interest to microwave device designers for some years. Undoped lithium ferrite, $Li_{0.5}Fe_{2.5}O_4$, has the highest Curie temperature (670°C) among the ferrimagnetic oxides (10). This factor, coupled with excellent square-loop properties, has resulted in a domination of the computer core industry by lithium ferrite compositions (See Greifer (11)), a situation analogous to the present position of garnets in the microwave industry. These same properties are also important for microwave devices: high squareness and remanence ratios for latching devices and wide temperature performance for most microwave devices. The high ratios of anisotropy to magnetostriction in lithium ferrites result in low stress-sensitivity of the remanence. Furthermore, lithium ferrites do not contain costly ingredients. Therefore, the potential exists for sizeable reductions in the costs of manufacturing microwave devices.

A number of problems, characteristic of a variety of lithium ferrite compositions, have prevented broad utilization of these materials. Probably the most important have been high dielectric and magnetic loss tangents. High anisotropy, high coercive forces, and low densities have also been instrumental in preventing these materials from reaching their potential.

1.2 Previous Studies

In one of the landmark investigations aimed at producing useful microwave materials, Jefferson and West (12) fabricated a series of lithium ferrites containing titanium and aluminum substitutions to provide a range of saturation magnetizations. Manganese, zinc, and nickel were among the other constituents which were explored. The resulting materials produced desirable phase-shift properties. However, the dielectric loss tangents were high (≥ 0.004), and no data were given for low-field magnetic loss, coercive force, or density.

Some years later, Vassiliev and Lagrange (13) reported the development of a group of lithium ferrites which were doped with aluminum and manganese. These materials covered a range of magnetizations from 1340 - 3770 gauss and had satisfactory squareness and remancence ratios for materials fired at 1200°C. Coercive forces varied from 1.7 to 2.5 Oe. Lower coercive forces (1.1 to 1.9 Oe) were obtained for compositions fired at 1250°C, but squareness and remanence ratios were low. Resonance linewidths were all greater than 489 Oe, and dielectric loss tangents were all greater than 489 Oe, and dielectric loss tangents were all in excess of 0.004. No low-field loss data were given.

In a study which formed the foundation for the results to be presented in this paper, Baba and Banerjee (14) explored a variety of lithium-aluminum and lithium-titanium ferrites. The first phase of this work was concerned primarily with lithium-aluminum compositions, and was later terminated because of poor microstructures and high coercive forces forces consistently found in that system. Efforts were then concentrated on lithium-titanium compositions which were easier to sinter and produced better microstructures. The compositions which resulted were based on substitutions of copper and manganese, and had promising phase-shifter properties. Preliminary magnetic loss measurements in a helical phase-shifter indicated loss levels approaching those of some garnets. However, dielectric loss tangents, coercive forces, and densities were in need of improvement.

A later study of lithium-titanium ferrites was performed by Collins and Brown (15) whose compositions covered a magnetization range of 2600 to 3600 gauss. Manganese substitutions were also used to achieve low dielectric losses, but oxygen annealing after firing was necessary to yield dielectric loss tangents less than 0.0005.

An investigation by Bunina et al. (16) was concerned with the effect of cobalt additions on the spinwave linewidths (ΔH_k) of a group of lithium-titanium ferrites. It was demonstrated that small quantities of cobalt dramatically increas ΔH_k in lithium-titanium ferrites. This result is in agreement with the independent findings reported below by the present authors. Bunina et al. further found that the ΔH_k of the cobalt-doped materials was strongly influenced by the presence of zinc.

2.0 MATERIAL REQUIREMENTS

The required properties for microwave materials, of course, vary in accordance with device applications. A wide variance can be expected depending on whether the material is used at resonance, off resonance, or in its own remanent field (latching devices). In the program reported here, the major interest was in latching devices, in particular, phase-shifters. For materials of this type, hysteresis loop properties are critical in addition to the need for good microwave behavior. In the ensuing discussion, the requirements for latching devices are accented. However, as will be seen in Section 5.0, low anisotropies (and resonance linewidths) are obtainable, thus the lithium ferrite family will impact a broad range of microwave devices. Table I gives a listing of the requirements established as goals for the investigation discussed in this report.

2.1 Magnetization ($4\pi M_s$)

The magnetization for a particular application is dictated by the frequency of operation in accordance with the principles discussed in the next section on magnetic loss. In Table I, the range of 200 to 2000 gauss is adequate for frequencies from L-band to well above C-band. The work reported here was concentrated mainly at 1000 gauss and below.

2.2 Magnetic Loss Tangent ($\tan \delta_m$)

Ince and Stern (17) found that the loss of a magnetic material in a digital phase shifter could be approximated from the low-field magnetic loss, or magnetic loss tangent, as well as the dielectric loss.

Table I. Goals for Materials

$4\pi M_s$	200 - 2000 G
$\tan\delta_m$	$\leq$ 0.001*
$\tan\delta_e$	$\leq$ 0.0002
ΔH_k	1 - 10 Oe
H_c	$\leq$ 1.0 Oe
$R = \frac{B_R}{4\pi M_s}$	$\geq$ 0.75
$S = \frac{B_R}{B \text{ at } 5 H_c}$	$\geq$ 0.90
$\frac{B_R(25^\circ C) - B_R(70^\circ C)}{B_R(25^\circ C)}$	Low as possible
Stress-sensitivity of B_R	Low as possible
Porosity	$\leq$ 1%

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*Measured at $\frac{\gamma(4\pi M_s)}{\omega} = 0.5$

As indicated in Table I, values of the magnetic loss tangent less than 0.001 are desirable.

According to principles set forth by Polder and Smit (18) and Lax and Button (19), domain resonance losses are avoided when the operating frequency, ω, is greater than the resonance cutoff frequency, $\omega_c = \gamma (H_A + 4\pi M_s)$ where H_A is the anisotropy field. Thus, one requirement for minimal low-field losses is

$$\frac{\omega_c}{\omega} = \frac{\gamma (H_A + 4\pi M_s)}{\omega} < 1$$

Even when the above relationship is observed, low-field losses do not vanish. Initial measurements of the magnetic loss (μ'') of a number of ferrites and garnets by Temme (20) using a coaxial line technique (21) indicated that the dominant parameter was ω_s/ω where $\omega_s = \gamma 4\pi M_s$. The lower limit of μ'' obtained by this technique was 0.005. By a more sensitive method, Green et al. (22) were able to extend the range of measurements to a μ'' of about 10^{-4}. Their data suggested the expression:

$$\mu''_{demag} = A \left(\frac{\omega_m}{\omega}\right)^N$$

where A and N are constants, and $\omega_m = \gamma (4\pi M_s)$. The constant A appears to be related to relaxation effects which is in qualitative agreement with other observers (23). Thus the presence of relaxing ions such as Gd^{3+}, Co^{2+}, etc. increases the magnetic loss.

The data in the studies discussed above were obtained for materials with low H_A, therefore experimental evidence is minimal relative to the effect of H_A on the magnetic loss. However, as seen in the criterion for domain resonance losses, a low H_A is desirable for device operation near ω_c. For devices which can be operated well above ω_c, H_A may be neglected in favor of better temperature performance and remanence behavior.

2.3 Dielectric Loss

The primary source of dielectric loss in ferrites at microwave frequencies is generally ascribed to conduction by means of electrons jumping from divalent iron ions on octahedral sites to neighboring trivalent iron ions on octahedral sites. In undoped stoichiometric lithium ferrite, $Li^{1+}_{0.5}Fe^{3+}_{2.5}O_4$, there are no divalent iron ions. Therefore, there should be no conduction, and the dielectric loss should be very low.

In the practice of sintering lithium ferrites, temperatures well above 1000°C are employed. It has been reported that above approximately 1000°C, the equilibrium oxygen pressure over lithium ferrite is greater than one atmosphere (24). Considering that lithium ferrites are typically fired at above 1150 to 1250°C to achieve porosities below 10%, oxygen dissociation and, additionally, lithia volatility are factors. As a result, divalent iron ions are generated. For a more detailed discussion of this process, see Ridgley et al.(25).

The dielectric loss of lithium ferrites can be orders of magnitude greater than the value of 0.0002 shown in Table I. One way to lower the loss is to decrease the number of divalent iron ions present by the well-known buffering reaction (26,27):

$$Fe^{2+} + Mn^{3+} \rightleftarrows Fe^{3+} + Mn^{2+} .$$

Because the equilibrium point is toward the right in this reaction, a small addition of trivalent manganese decreases the divalent iron level.

A second way to lower the loss is to find a way of achieving adequate densification at low firing temperatures. The method found to accomplish this goal was perhaps the major contribution of the program reported here, and will be discussed in Section 3.6.

2.4 Spinwave Linewidth (ΔH_k)

To accommodate a broad range of power-handling capabilities, Table I indicates a range of ΔH_k values from 1 to 10 Oe. ΔH_k is easily increased by the addition of relaxing ions. However, as indicated in Section 2.2, the magnetic loss is also increased.

2.5 Hysteresis and Stress-Sensitivity Properties

The properties of interest are the remanence ratio ($R = B_R / 4\pi M_s$), the coercive force (H_c), and the squareness ratio ($S = B_R/B$ at $5\ H_c$). As previously discussed, high values of R and S are common in the lithium ferrite system. However, lithium ferrites typically have H_c values above 2 Oe bacause of high anisotropy fields, excessive porosity, and small grain size. To compete favorably with garnets in latching devices, values ≤ 1 Oe are necessary.

Under conditions in which there is a close mechanical fit between a ferrite part and a device housing, stresses may be generated in the ferrite (23) , particularly where thermal gradients are present. For an independent grain model, Dionne (2) has shown that the effect on R of uniaxial stress parallel to the magnetic field is determined primarily by the ratio of the hard-axis magnetostriction constant to the anisotropy constant. A later analysis (29), more analogous to a polycrystalline structure, indicated that both magnetostriction constants, λ_{111} and λ_{100}, are involved, but the hard-axis constant is dominant. The combination of magnetostriction constants that are reduced by the addition of manganese (3) and inherently high anisotropy leads to a low ratio of magnetostriction to anisotropy, desirable for low stress-sensitivity of remanence in the lithium ferrites.

For the case of internal stresses, models proposed by Smit (1) and Weisz (42) indicate that the easy-axis constant (λ_{111}) is important in determining the remanence ratio. Since $\lambda_{111} << \lambda_{100}$ for the lithium ferrite family (3), this property also may contribute to high values of R and S.

2.6 Temperature Coefficient of Remanence

As shown in Table I, the temperature range of interest for this program was 25 to 70°C. In spite of the high degree of nonmagnetic doping required to bring the magnetization down to the specified range, the lithium ferrites have adequate Curie temperatures. In comparison, the gadolinium - doped garnets also perform well in this respect because of their compensation points below room temperature.

2.7 Porosity

A high degree of porosity in a microwave material is undesirable from several standpoints. Where hysteresis loop properties are important, such as in latching devices, porosity has a deleterious effect on both R (30) and H_c (31). The porosity also has a negative influence on tan δ_m (32) and allows moisture absorption, which increases tan δ_e. Also of significance is the fact that a porous material is usually difficult to fabricate reproducibly, leading to fluctuations in the dielectric constant.

Lithium ferrites have traditionally been difficult to density sufficiently. Oxygen dissociation, lithia volatility, and discontinuous grain growth have placed a limitation on the sintering temperatures which may be employed. At the 1150 - 1250°C sintering range which is typical for these materials, porosities are generally about 5 to 10%. Porosities as low as 1% at firing temperatures near 1000°C were achieved in this program. The technique by which these features were accomplished is discussed in Section 3.6.

3.0 COMPOSITIONAL CONSIDERATIONS

The formulations for effective lithium ferrite compositions became quite complex as the program progressed. In addition to the lithium and iron in the basic formula, up to six other cationic constituents have been employed. Each constituent has been included for one or more specific purposes.

The effects of each constituent are summarized in Table II. The notations of Beneficial, Minimal Effect, and Deleterious should not be considered generally applicable. Some of the constituents may behave differently in other ferrite systems. Furthermore, the effects listed in Table II are pertinent only for the quantities in which they were incorporated into the formulations in the present investigation. For example, a constituent may be beneficial to the remanence ratio when used in small quantities, but may degrade it in large quantities.

3.1 Titanium

The primary function of the titanium is to lower $4\pi M_s$. Undoped stoichiometric lithium ferrite, $Li_{0.5}Fe_{2.5}O_4$, has a $4\pi M_s$ of approximately 3730 gauss (25). As in all ferrites, the magnetization may be reduced by the substitution of various nonmagnetic ions for iron ions in the octahedral sublattice (33).

Because aluminum ions are known to have a strong affinity for octahedral sites, aluminum is often employed in ferrites to lower the magnetization. In the initial phases of our program, attempts to densify

Table II. Effects of Constituents

	Titanium	Zinc	Manganese	Cobalt	Nickel	Bismuth
Tanδ_m	B	B	M	D	M	M
Tanδ_e	M	M	B	M	M	B
$4\pi M_s$	B	D	M	M	M	M
ΔH_k	M	B/M	M	B	M	M
H_c	D	B	B	D	M	B
R and S	D	D	B	D	B	B
Temperature Coefficient	D	D	D	M	M	M
Stress-Sensitivity	M	M	B	M	M	M
Porosity	D	B	B	M	M	B

B = Beneficial
M = Minimal Effect
D = Deleterious

aluminum compositions required sintering temperatures which gave high losses, and the resulting microstructures were never satisfactory. Resonance linewidths also increased with increasing substitutions of aluminum. This effect may be explained partly by the porosity and poor microstructure, and partly by an increasing anisotropy field. Yakovlev et al. (34) found that aluminum substitutions in lithium ferrite decrease the magnetization to a greater extent than the anisotropy. Thus, the anisotropy field increases with the addition of aluminum.

Because of the problems associated with aluminum substitutions, other nonmagnetic octahedral substitutions were explored. Titanium, which also has been explored by other investigators (12, 15, 16, 35), yielded the best combination of properties. Titanium gave better microstructures, resulting in lower coercive forces and linewidths than were obtainable in the aluminum-substituted materials. The principal drawback in using titanium is the requirement for compensation of its high valence state; i.e., 4+ compared with 3+ for aluminum. If not properly compensated, the titanium causes iron to become divalent, with a concomitant sharp increase in losses. In this study, the titanium was compensated in part by divalent ions and by additional monovalent lithium. Since lithium ions in excess of approximately 0.5 ions/formula unit preferentially populate tetrahedral sites (35), the net result of such compensation is substantial lowering of the Curie temperature and a greater temperature coefficient.

As for the other items in Table II, the effects of titanium are less important. The beneficial decrease of $\tan \delta_m$ is mainly attributable to the decrease of $4\pi M_s$. The deleterious effects on H_c, R, S, and porosity are not serious.

3.2 Zinc

Zinc performs a number of beneficial functions. One of the most important is the lowering of the anisotropy field. Therefore, zinc aids in meeting the Polder-Smit magnetic loss criterion discussed above, and it decreases the resonance linewidth. It also provides a two-fold effect in controlling H_c. Besides decreasing the anisotropy, zinc has long been known as a fluxing agent in ceramic systems. It thereby promotes densification and grain growth, both of which decrease H_c. Zinc also increases ΔH_k in cobalt-doped systems as shown by Bunina et al. (16). The effect on ΔH_k is minimal when cobalt is not present.

Some deleterious effects of zinc arise as a consequence of the preferential tetrahedral site occupancy. Such occupancy by nonmagnetic ions increases $4\pi M_s$, offsetting the effect of the titanium, and causes a large drop in the Curie temperature . Zinc also decreases the squareness and remanence ratio.

3.3 Manganese

Manganese is added for two reasons. Because of the buffering reactions discussed earlier, tan δ_e is diminished. Moreover, it has been shown that manganese ions can reduce the magnetostriction constants in lithium ferrite (3). As a result, the stress-sensitivity is lowered, and as a by-product, R and S are improved. Manganese is also somewhat beneficial as a sintering aid, thereby diminishing porosity and H_c. A decrease in the Curie temperature is the only negative factor associated with the addition of manganese.

3.4 Cobalt

Divalent cobalt ions on octahedral sites are relaxing ions, and very small quantities broaden the spinwave linewidth. By the same token, an increase in tan δ_m must be tolerated. Other effects of cobalt substitutions are an increased H_c and a slight degradation of S and R, possibly as a result of increased magnetostriction. Interestingly, cobalt does not produce a minimum in anisotropy or resonance linewidth in the lithium-titanium ferrite system. This effect has been noticed by Jefferson and West (12) and has been explained by Banerjee et al.(36).

3.5 Nickel

Small quantities of nickel enhance the squareness and remanence ratio of lithium ferrite as reported by West (37). Other properties are minimally affected.

3.6 Bismuth

The key to the low tan δ_e, H_c, and porosities, all of which will be demonstrated in Section 5.0, is the addition of minute quantities of bismuth. Bismuth oxide is liquid above 825 °C and forms low-melting eutectics in a variety of oxide systems. By means of this additive, densities > 99% are obtainable at firing temperatures near 1000 °C. Avoidance of high firing temperatures minimizes the oxygen loss and lithia volatility discussed in Section 2.3, thereby reducing the occurrence of divalent iron and allowing low values of tan δ_e.

4.0 EXPERIMENTAL PROCEDURES

4.1 Sample Preparation

Conventional ceramic procedures are followed in the preparation of the lithium-titanium ferrites. Reagent grade oxides and carbonates are weighed and mixed in steel ball mills with steel balls. The resulting slurry is dried, crushed, and then placed in refractory trays. The material is then calcined at temperatures of 700°C to 900°C. This particular operation is not a critical one over the range of temperatures given. The ferrite powder is milled once again in steel mills, and is then dried, granulated, and screened. An isobutyl-methacrylate binder is added to the powder, and the material is suitably granulated for pressing in a steel die.

For evaluations, cylindrical and toroidal samples are pressed and fired. Typically, sintering temperatures are concentrated in the 950-1050°C range for optimum properties. Emphasis is placed on keeping the sintering temperature as low as possible, yet consistent with good density. Samples are fired in air or oxygen, but the latter atmosphere leads to lower dielectric loss. After firing, samples are ground to a number of shapes (spheres, discs, etc.) for various measurements.

4.2 Measurements

Saturation magnetization and Curie temperature are measured on spherical samples in a vibrating sample magnetometer equipped with a sample heater. The hysteresis loop properties are obtained from toroidal samples by means of a 60 Hz B-H loop tracer which employs an operational amplifier in the integrating circuit, which was designed to minimize phase shift problems. The resonance linewidth is measured at X-band or at S-band using essentially the ASTM method. For the dielectric constant, dielectric loss, permeability, and magnetic loss, a resonant post technique described by Courtney (38) is employed. The anisotropy field ($2|K_1/M_s|$) is determined from the approach to saturation method reported by Dionne (39). The spinwave linewidth (ΔH_k) is obtained simply by weighing a cylinder whose dimensions are measured. When a more accurate value is desired, the displacement method is used to determine the volume.

5.0 RESULTS

5.1 Material Properties

In Fig. 1, $4\pi M_s$ and Curie temperature data are given for a series of lithium ferrites in which aluminum and titanium have been substituted for iron. Also included in the figure are data points taken from studies by Vassiliev (41) and Blasse (35). Other values of magnetization in the literature were not included because sufficient information was not given to convert the units to gauss. The significant conclusion to be drawn from Fig. 1 is that equal substitutions of aluminum or titanium give almost the same $4\pi M_s$ values. Titanium substitutions yield slightly higher $4\pi M_s$ values and substantially lower Curie temperatures at values of $x > 0.4$ ions per formula unit. The Curie temperature behavior is readily understood through an examination of the formulas for the aluminum and titanium series, $Li_{0.5}Fe_{2.5-x}Al_xO_4$ and $Li_{0.5+0.5x}Fe_{2.5-1.5x}Ti_xO_4$. The additional 0.5 x lithium ions per formula unit in the titanium series are added to compensate for the quadrivalence of the titanium ions $(1.5\ Fe^{3+} = Ti^{4+} + 0.5\ Li^{1+})$.

Blasse (35) found that lithium ions in excess of 0.5 ions/formula unit populate tetrahedral sites. These tetrahedral nonmagnetic ions weaken the exchange interaction and decrease the Curie temperature beyond the contribution of the octahedral titanium.

When zinc ions are added, they also enter the tetrahedral sites, and their behavior is similar to that of the excess lithium ions.

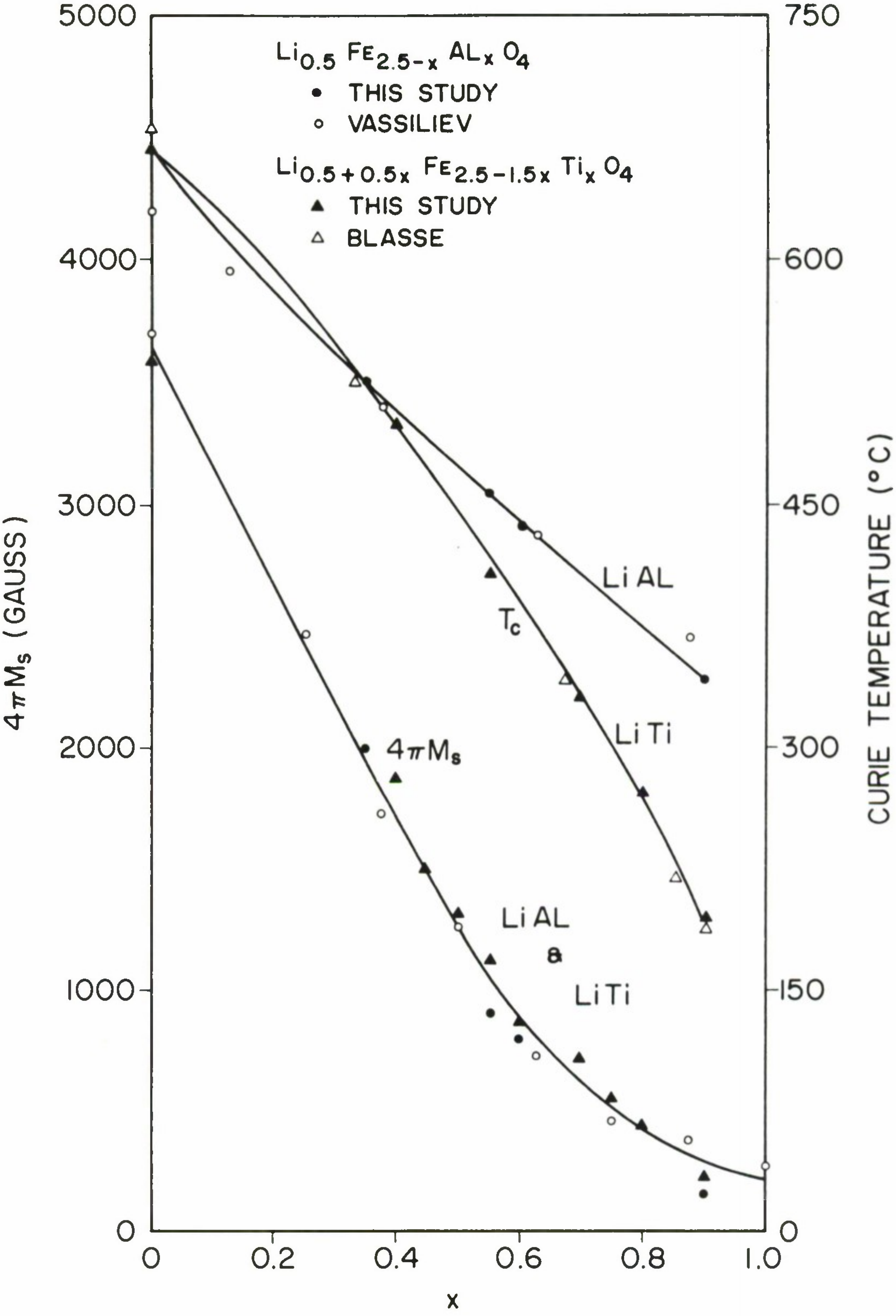

Fig. 1 Room temperature saturation magnetization and Curie temperature as a function of aluminum and titanium substitution in lithium ferrite.

Figures 2 and 3 show the effect of zinc on the $4\pi M_s$ versus temperature curves for two representative values of magnetization, 1000 and 400 gauss. In both figures, a series of three lithium ferrites with varying zinc levels is compared to the appropriate yttrium-aluminum and yttrium-gadolinium-aluminum garnets. Normally, an increase of $4\pi M_s$ is expected with the addition of a nonmagnetic ion such as zinc on the tetrahedral site. To allow identical $4\pi M_s$ values at room temperature, the addition of zinc has been offset by adjusting the titanium content slightly for both the 1000- and 400-gauss series. As expected, the zinc causes a sharp drop in the Curie temperature, so the amount of zinc to be employed should be approached with caution.

Zinc also exerts an influence on the hysteresis properties. In Fig. 4, a group of hysteresis loops for a series of 1000-, 600-, and 400-gauss compositions is presented. At each magnetization level, three variations of zinc content are shown. In order that visual comparisons of loop shapes may be performed easily, each loop is normalized to the same B_{max} and H_c. Values of H_c and S are given below each loop. As expected, H_c decreases as zinc is added, consistent with the decrease of anisotropy and the fluxing action discussed previously. Moreover, the squareness also diminishes with increasing zinc content, probably because of a declining domination of anisotropy energy over magnetostrictive energy in the [111] direction. The effect of titanium on the loops may also be seen in Fig. 4. As titanium is increased from the 1000-gauss compositions to the 400-gauss compositions, H_c rises and S declines.

Another important factor which influences the coercive force and other properties is the porosity. Reduction of porosity, or maximization of the density, in lithium ferrites has been a problem for a considerable time. To overcome this problem, a number of fluxes were investigated leading to the utilization of the highly reactive additive, bismuth.

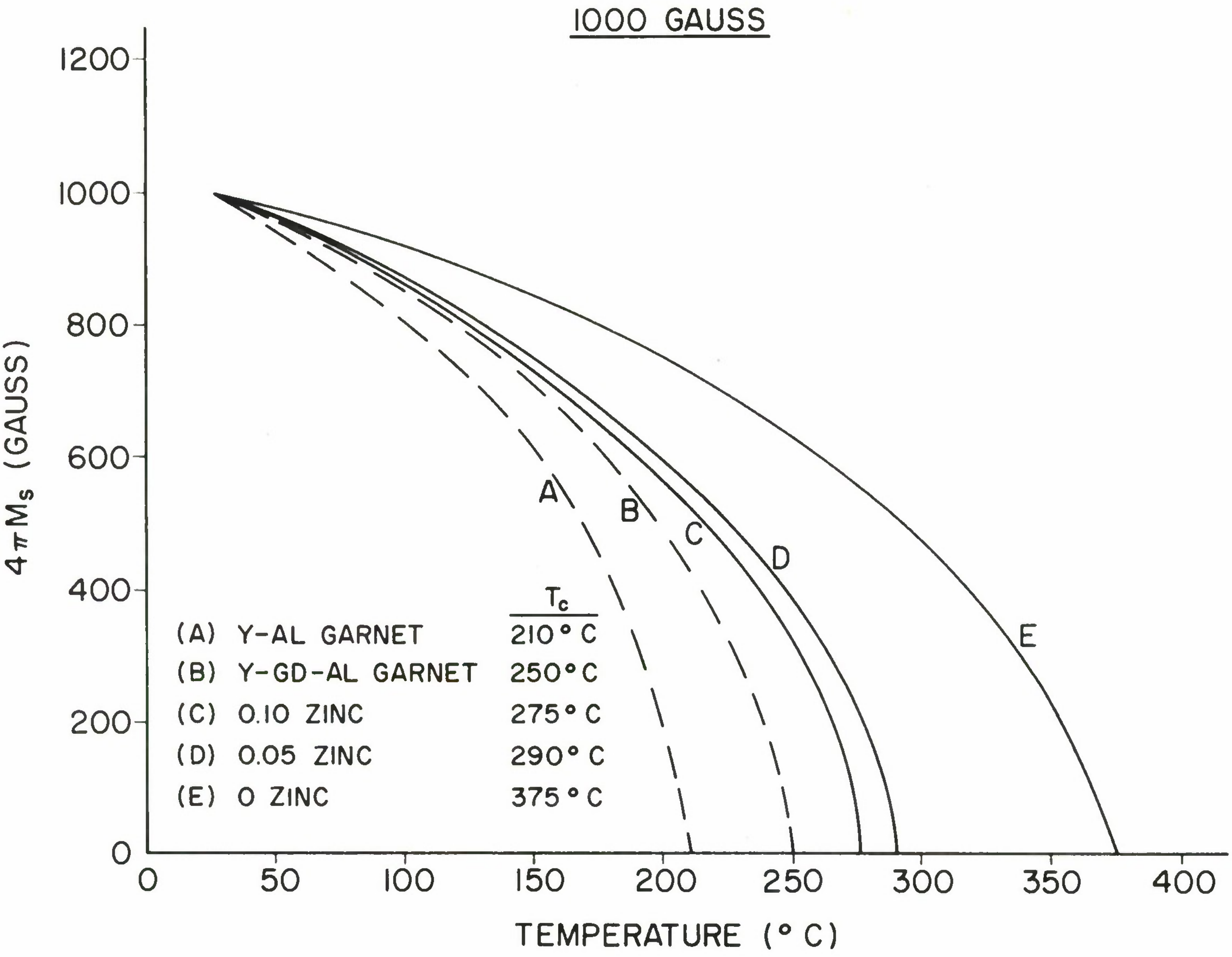

Fig. 2 Saturation magnetization versus temperature for a group of lithium ferrites and garnets with room temperature values of 1000 gauss.

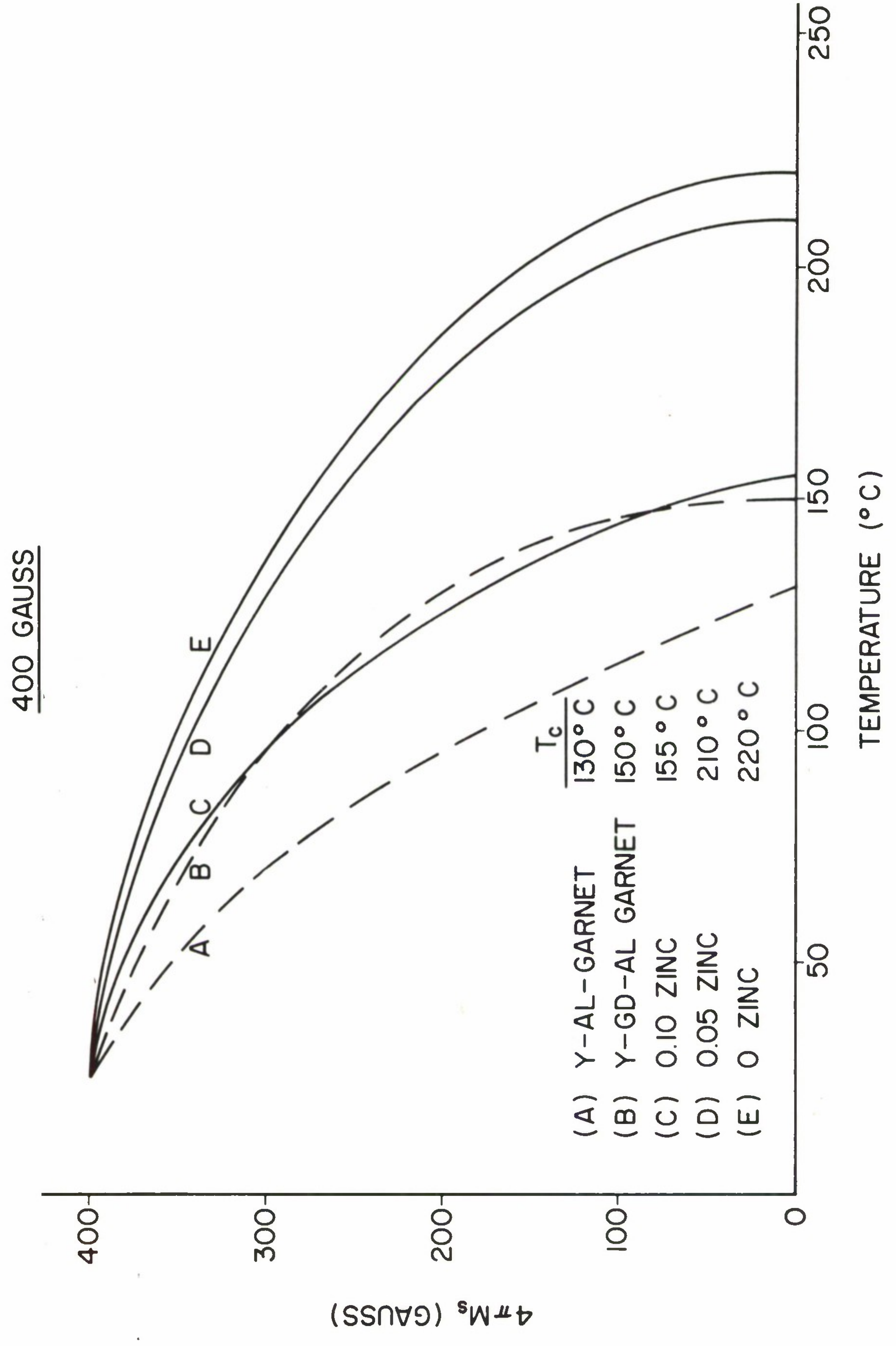

Fig. 3 Saturation magnetization versus temperature for a group of lithium ferrites and garnets with room temperature values of 400 gauss.

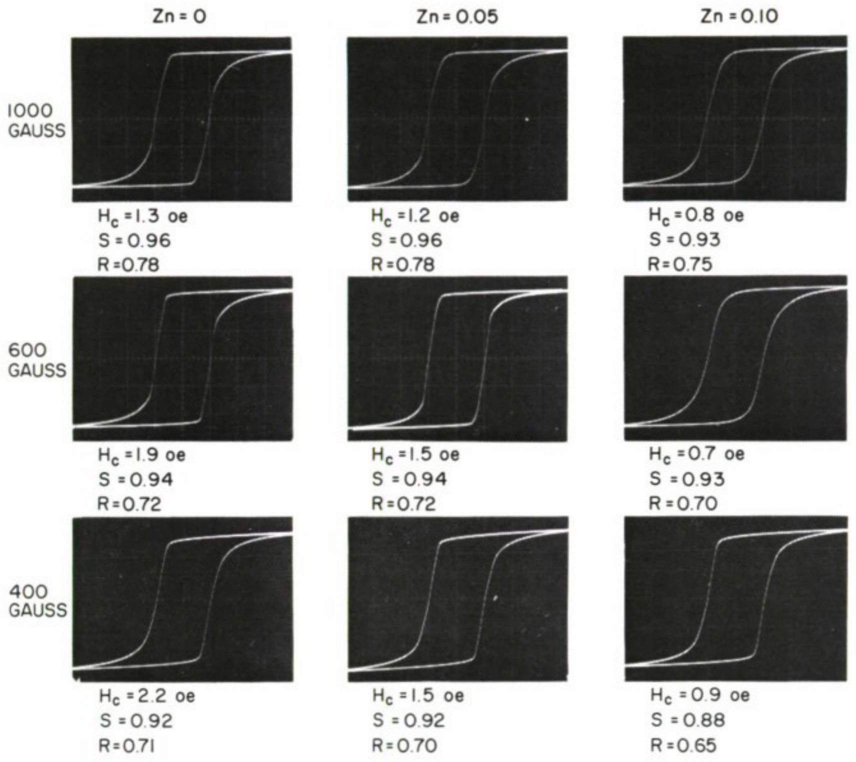

Fig. 4 Hysteresis loops for lithium ferrites with three different $4\pi M_s$ levels and varying zinc content. All loops are normalized to the same B_{max} and H_c values so that shapes may be readily compared.

The effect of this additive on the densification is shown in Fig. 5 for the composition $Li_{0.8}Zn_{0.1}Bi_xFe_{1.4-x}Ti_{0.7}O_4$. The powerful fluxing action of bismuth is demonstrated by the small quantities which yield densities near x-ray density. At 1100°C, as few as 0.0005 atoms/formula unit of bismuth are sufficient for approaching within a few percent of x-ray density; while the control sample, without bismuth, is only about 84% of x-ray density. At the optimum firing temperature for the control composition, 1200°C, a density of 91% is attained (not shown in figure). In contrast, the compositions with 0.0005 and 0.001 ions per formula unit of bismuth yield substantially higher densities over the range of temperatures presented. The most significant feature of the densification behavior is that densities of approximately 99% are obtained for x values of 0.002 and 0.005 at temperatures as low as 1000°C. By firing the material at low temperatures, dielectric loss problems are minimized.

In Fig. 6, additional evidence of the densifying action of bismuth is seen. Figure 6A is a photomicrograph of the control composition fired at 1200°C; Fig. 6B represents the same material with 0.002 ions/formula unit of bismuth added and a firing temperature of 1000°C. The surfaces in Figs. 6A and 6B are both lapped but unetched. The superior character of the bismuth-containing material is dramatic in spite of a 200°C lower firing temperature. In Fig. 7, the bismuth-doped material has been lapped and thermally etched to show the grain size. The high degree of surface texture is common to lithium-titanium ferrites as a result of either chemical or thermal etching. The micrograph illustrates the large-grained character of the bismuth-doped materials. Most grains are above 100 μm in diameter, an important factor for achieving low H_c values.

The effect of bismuth on H_c is clearly demonstrated in Fig. 8. The composition without bismuth has $H_c > 2$ Oe for firing

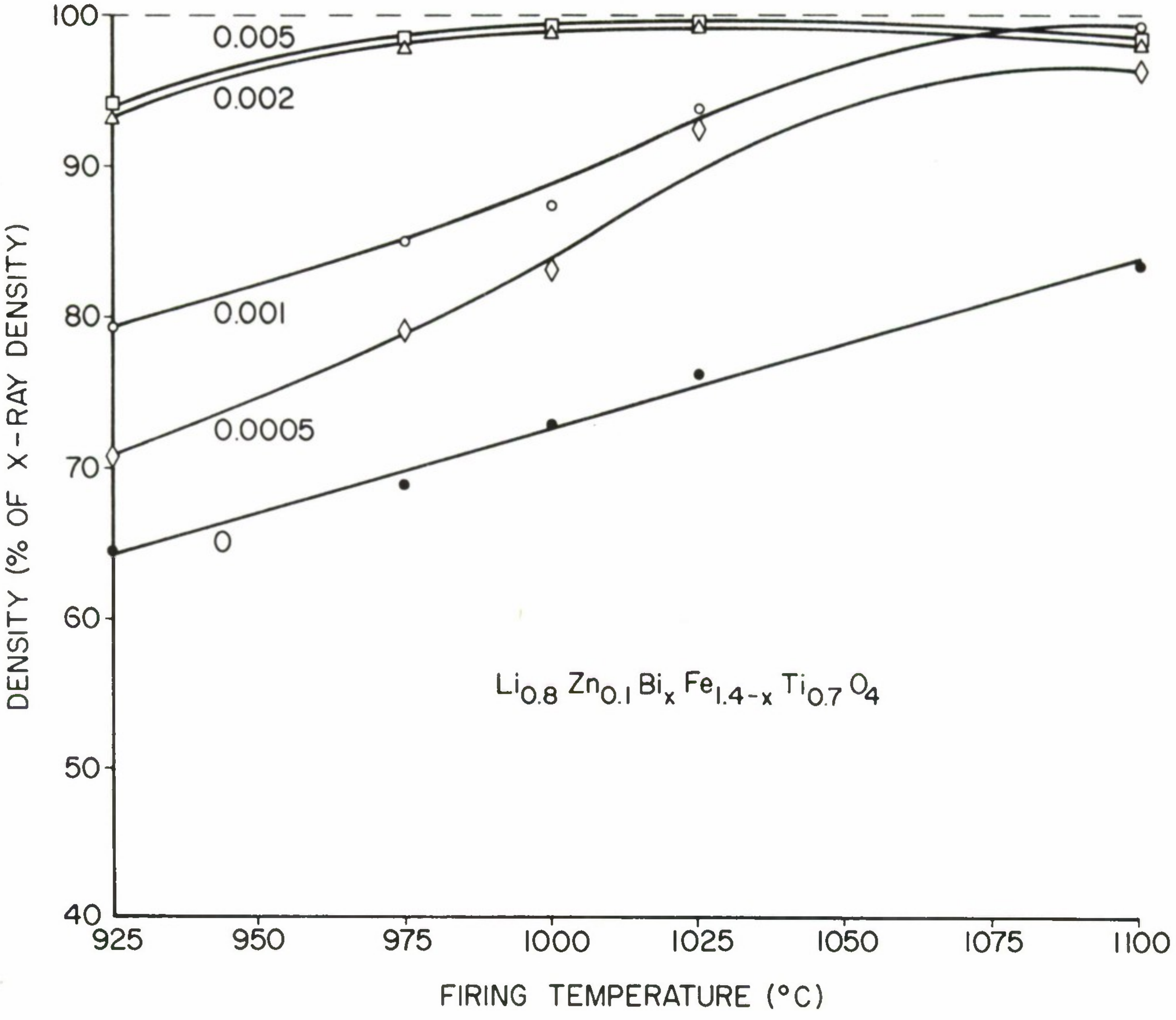

Fig. 5 Density versus firing temperature for $Li_{0.8}Zn_{0.1}Bi_xFe_{1.4-x}Ti_{0.7}O_4$

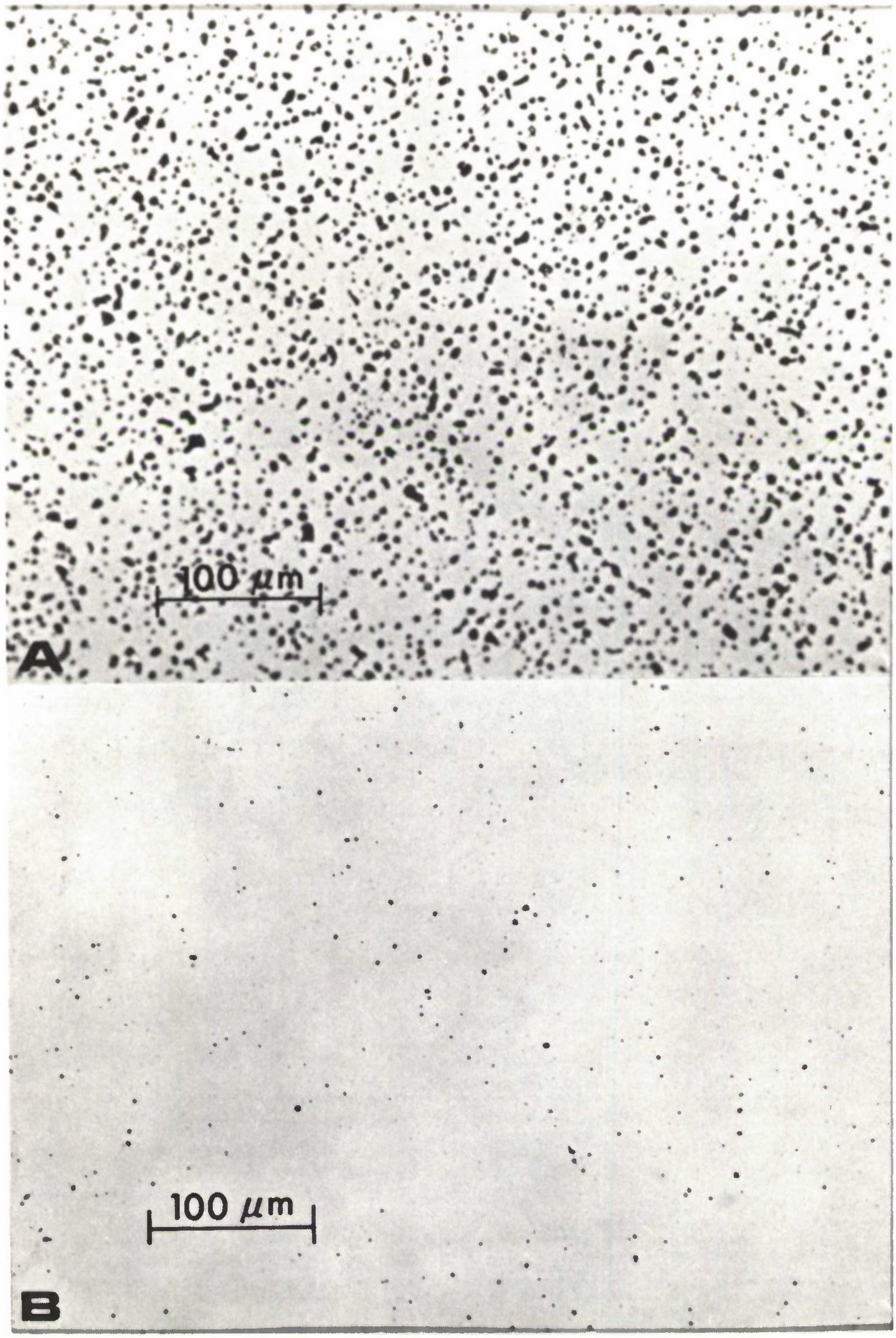

Fig. 6 Polished unetched samples of lithium ferrite having the same basic composition except for bismuth content. (A) No bismuth, fired at 1200°C; (B) Bismuth = 0.002 ions/formula unit, fired at 1000°C.

Fig. 7 Polished and etched sample of lithium ferrite with bismuth = 0.002 ions/formula unit.

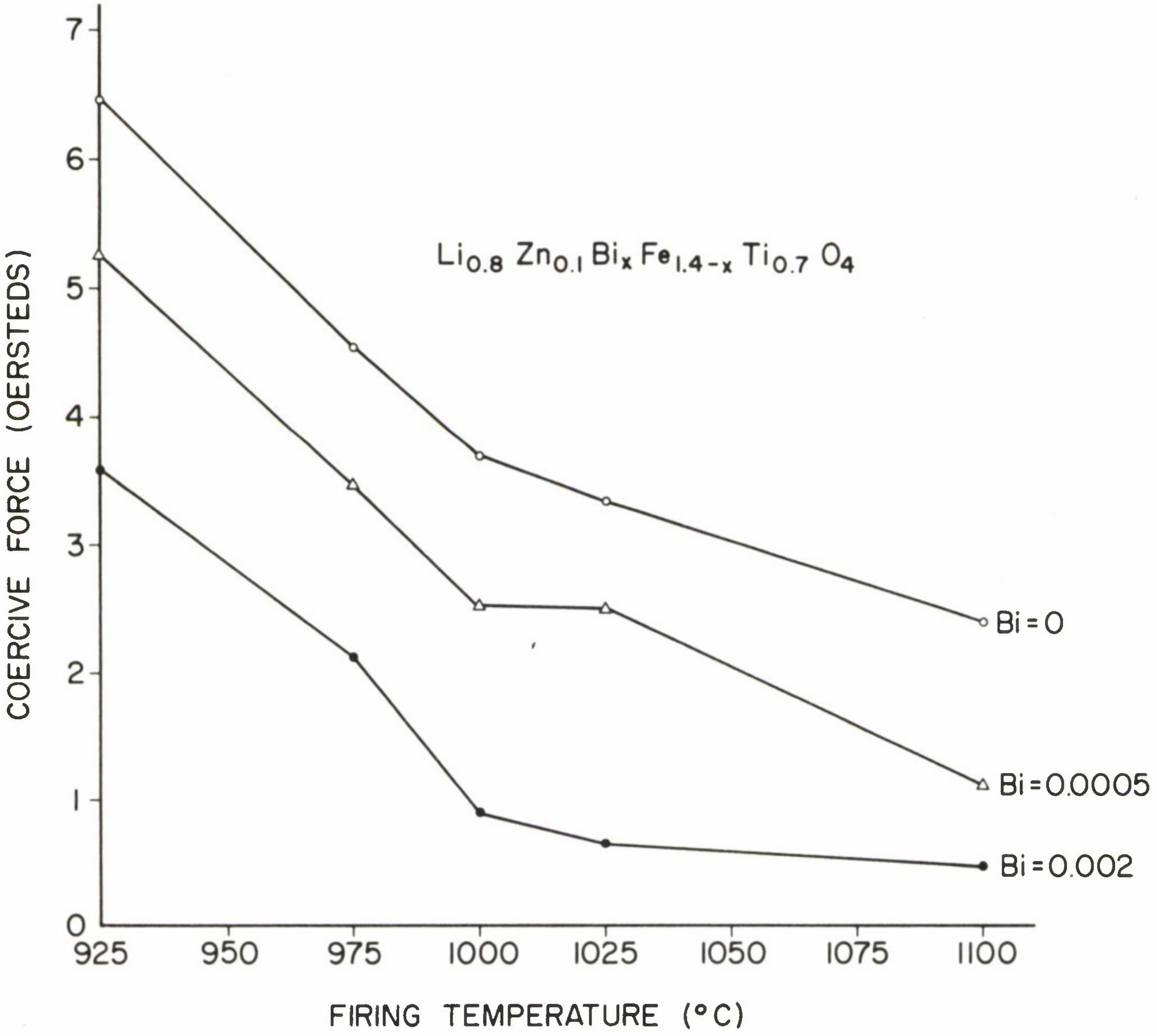

Fig. 8 Coercive force versus firing temperature for $Li_{0.8}Zn_{0.1}Bi_xFe_{1.4-x}Ti_{0.7}O_4$.

temperatures up to 1100°C. Significant reductions are obtained by the addition of small quantities of bismuth as illustrated in the figure. For the composition with 0.002 ions/formula unit of bismuth, values less than 1 Oe are typical when firing temperatures are 1000°C or above.

To allow a suitable range of power-handling capabilities, small quantities of cobalt were explored. In Fig. 9, the effect of cobalt on ΔH_k, tan δ_m, and tan δ_e are presented for the composition $Li_{0.78-0.5x}Zn_{0.1}Co_xMn_{0.05}Fe_{1.408-0.5x}Ti_{0.66}Bi_{0.002}O_4$. For quantities of cobalt up to 0.01 ions/formula unit, the relationship is linear, and ΔH_k may be varied from 2.5 to 8.3 oersteds. The improvement in the peak power-handling capability is attained at the expense of the low-field magnetic loss. The magnetic loss tangent is increased by a factor of two when 0.01 cobalt ions/formula unit are added to the control composition. The effect of cobalt on tan δ_e is minimal. The close relationship between tan δ_m and ΔH_k is demonstrated in Fig. 10.

5.2 Representative Compositions

Each of the various ions which is incorporated in the lithium-titanium ferrite system is added to enhance primarily one or two properties. However, many of the additives deleteriously affect other properties to varying degrees. Thus it is necessary to examine compositions from the standpoint of the total combination of properties for microwave devices. In Table III, the compositional, firing, magnetic, physical, and microwave data are presented for 12 representative lithium-titanium ferrites. With the exception of ferrite H, which contains nickel, the main compositional differences are:

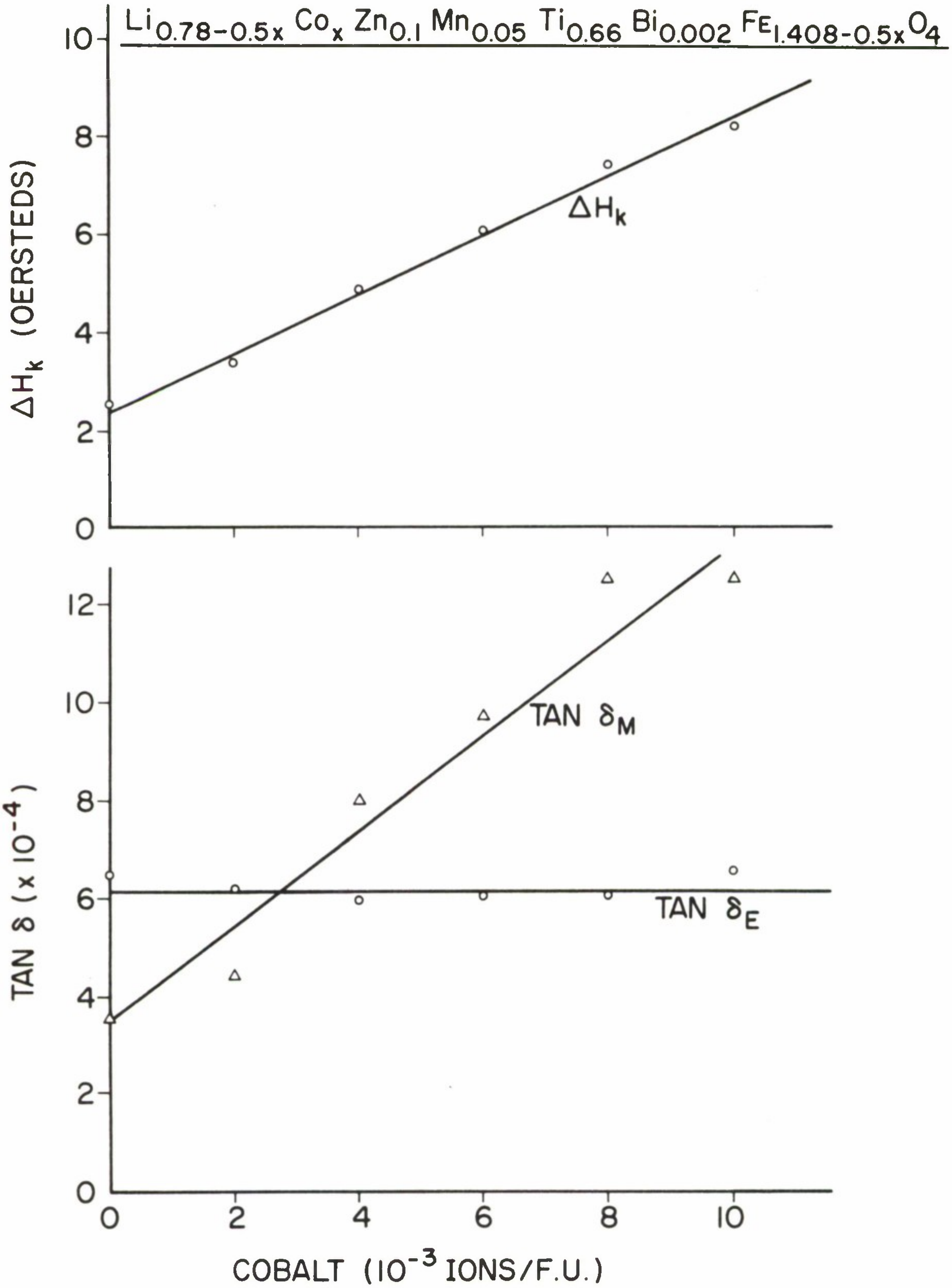

Fig. 9 Spinwave linewidth, tan δ_m, and tan δ_e for $Li_{0.78-0.5x}Co_xZn_{0.1}Mn_{0.05}Ti_{0.66}Bi_{0.002}$ $Fe_{1.408-0.5x}O_4$.

Table III. Representative Lithium Ferrites

	Li	Zn	Co	Fe	Ti	$4\pi M_s$ (gauss)	K/M_s (Oe)	H_c (Oe)	R	ΔB_R (%)	T_c (°C)	P (%)	ΔH^* (Oe)	ΔH_k^{**} (Oe)	$\tan\delta_m$ (10^{-4})	$\frac{\gamma(4\pi M_s)}{\omega}$	$\tan\delta_e$ (10^{-4})	ϵ'
A	.777	.10	.006	1.405	.66	1000	105	1.0	.70	11	275	1	210	6.0	8.0	.33	4.2	18.7
B	.780	.10	0	1.408	.66	1000	114	0.8	.70	11	275	1	210	2.3	4.6	.33	4.4	18.9
C	.750	.25	0	1.198	.75	1000	51	0.4	.50	26	170	1	50	3.5	5.6	.33	3.4	18.8
D	.775	.25	0	1.123	.80	750	46	0.4	.57	37	145	1	50	4.0	3.6	.24	4.5	19.3
E	.852	0	.006	1.380	.71	600	112	2.3	.74	6	290	2	320	4.8	5.4	.19	3.3	18.0
F	.847	.05	.006	1.295	.75	600	92	1.5	.73	13	235	1	260	5.5	6.7	.19	3.1	18.2
G	.847	.10	.006	1.195	.80	600	75	0.9	.70	19	195	1	160	7.4	5.8	.18	3.6	18.6
H	.823	.12	.004	1.131	.82	550	81	0.6	.70	26	175	1	100	6.7	5.0	.19	3.8	19.1
I	.893	0	0	1.268	.79	400	90	2.2	.76	10	220	2	260	1.8	3.8	.11	2.5	18.7
J	.885	.05	0	1.190	.82	400	77	1.5	.73	19	210	3	160	3.0	2.6	.12	2.7	18.7
K	.885	.10	0	1.093	.87	400	63	0.9	.67	33	155	3	100	3.5	3.1	.20	2.6	19.2
L	.875	.15	0	1.023	.90	400	35	0.3	.5	60	120	2	45	5.0	3.0	.12	3.5	19.3

* At 2.9 GHz
** At 5.8 GHz

All compositions contain .05 Mn ions/formula unit

Composition H contains .05 Ni ions/formula unit

Compositions I-K contain .005 Bi ions/formula unit; all other compositions contain .002 Bi ions/formula unit.

Legend: $4\pi M_s$ = saturation magnetization; K = anisotropy constant; H_c = coercive force;
R = remanence ratio; ΔB_R = % change in remanence between 25 and 70°C; T_c = Curie temperature;
P = porosity; ΔH = resonance linewidth; ΔH_k = spin wave linewidth; $\tan\delta_m$ = magnetic loss tangent;
γ = gyromagnetic ratio; ω = operating frequency; $\tan\delta_e$ = dielectric loss tangent; ϵ' = dielectric constant.

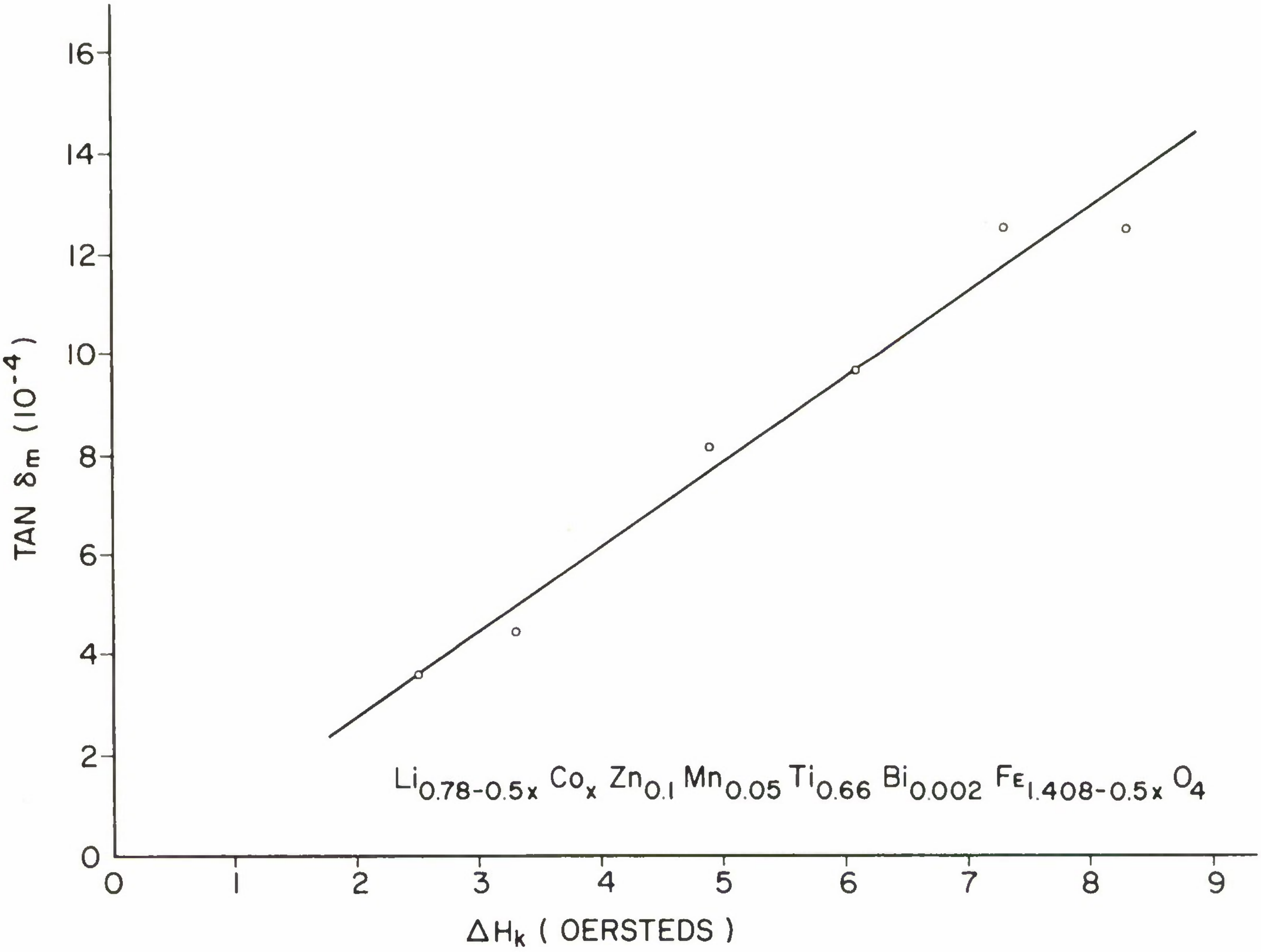

Fig. 10 Tan δ_m as a function of ΔH_k for $Li_{0.78-0.5x}Co_xZn_{0.1}Mn_{0.05}Ti_{0.66}Bi_{0.002}$ $Fe_{1.408-0.5x}O_4$.

(1) titanium-iron ratio to control $4\pi M_s$,
(2) zinc content to control anisotropy, and
(3) cobalt content for power-handling capability.

The compositions fall roughly into two categories. All compositions have good remanence ratios and Curie temperatures except C, D, and L. Thus, they are well-suited for latching device applications. C, D, and L have increased zinc contents to allow utilization where narrow resonance line widths are required. The large number of compositions in the table are presented primarily to demonstrate that considerable latitude exists for altering properties for special requirements. Moreover, a number of general conclusions may be drawn from the properties shown for the materials.

For instance, all of the $\tan \delta_e$ values are less than 0.0005, and some are below 0.0003. These values would probably be a little lower if the ASTM method were employed. The resonant post technique (38) is known to give slightly higher values than the ASTM technique. The table also shows that a wide range of coercive forces are obtainable without significant degradation of R. However, if a low H_c is required, the temperature sensitivity is adversely affected. Another point of importance for microwave device designers is the high dielectric constant which is typically found in the lithium-titanium ferrites. The dielectric constant of these materials is about 50% higher than typical microwave garnets and ferrites. A high dielectric constant is characteristic of materials containing titanium (e.g., TiO_2, $BaTiO_3$), and should provide additional latitude in new device designs.

Because of experimental limitations, Measurements of $\tan_m$ at levels of $\gamma(4\pi M_s)/\omega$ near 0.5 were not possible. However, measurements were made at levels < 0.5 and these data are shown in the table.

5.3 Device Performance

Figure 11 shows some performance characteristics of latching ferrite phasers made with selected lithium ferrites, and includes some results for garnet and magnesium-manganese ferrite phasers for comparative purposes. In general, the results obtained at C-band with selected lithium ferrites are as good or better than those obtainable with the garnet. In one case, not shown here, a material without any cobalt gives a figure of merit in excess of 700. Peak power-handling capability in this case is considerably less, but is comparable to a garnet with similar low loss characteristics. It is estimated that these materials will have wide use at C-band and above because of their good temperature characteristics and the lower cost of the lithium ferrite materials as compared to the garnets. At S-band the performance of a lithium ferrite is not quite as good as the garnet because of higher magnetic loss. This situation is presented graphically in Fig. 12. The curves for the C-band lithium ferrite (1000 gauss) and a gadolinium-aluminum garnet with an approximately equivalent ΔH_k show low losses relative to the data for the S-band lithium ferrites. However, the S-band lithium ferrites are still attractive in low average power applications, in which the reduced cost of these materials may offset the higher insertion loss cost (i.e., the cost of generating the extra RF power to overcome the higher loss). For the very high average power applications where loss is of greater importance, the garnets are preferred at the present time. Whether the magnetic loss characteristics of the high-power S-band lithium ferrites can be improved such that they are competitive with the comparable garnets is under investigation.

Circulators utilizing lithium ferrites have also been made. An L-band circulator had about a 10% greater loss than a garnet with an equivalent peak-power capability. However, if temperature stability is very important, as it may be in some applications for low-power fixed-field devices, the better temperature stability of the materials without zinc can be of great interest.

	S-Band			C-Band		
	MnGdAl Garnet	LiTiZn Ferrite	LiTiZn Ferrite	MgMn Ferrite	MnGdAl Garnet	LiTiZn Ferrite
$4\pi M_s$(G)	680	440	570	1000	1000	1000
f(GHz)	2.8	2.8	2.8	5.6	5.6	5.6
B_r(G)	470	300	400	530	635	700
H_a(Oe)	80	155	150	50	80	145
H_c(Oe)	0.7	1.5	0.9	0.5	0.7	1.0
ΔH_k(Oe)	6.5	3.2	7.8	5.8	6.0	6.3
ϵ'_d	30	30	38	16	16	16
w_f(in)	0.125	0.140	0.140	0.100	0.100	0.075
w_d(in)	0.100	0.080	0.080	0.030	0.040	0.040
w_g(in)	0.800	0.800	0.800	0.800	0.800	0.630
$\Delta\Phi$/in	70	55	70	60	75	85
FM(deg/dB)	650	500	400	500	550	500
P_p(kW)	5.7	4.9	5.8	-	7.0	-
P_{av}(W)	350	-	300	50	200	-

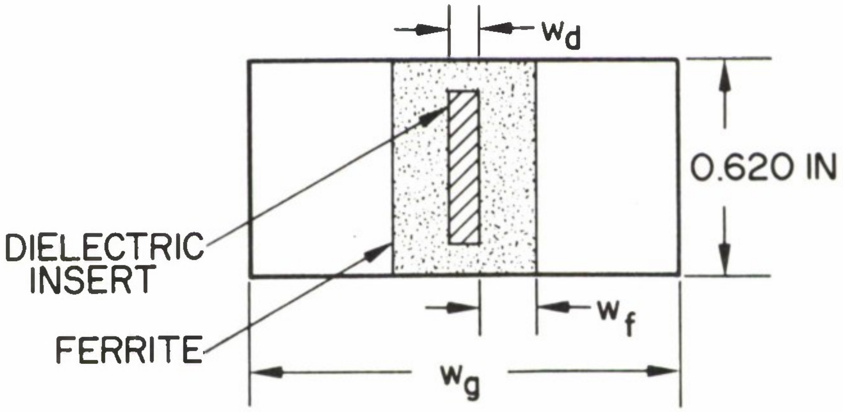

Fig. 11 Properties of interest for S-band and C-band microwave devices.

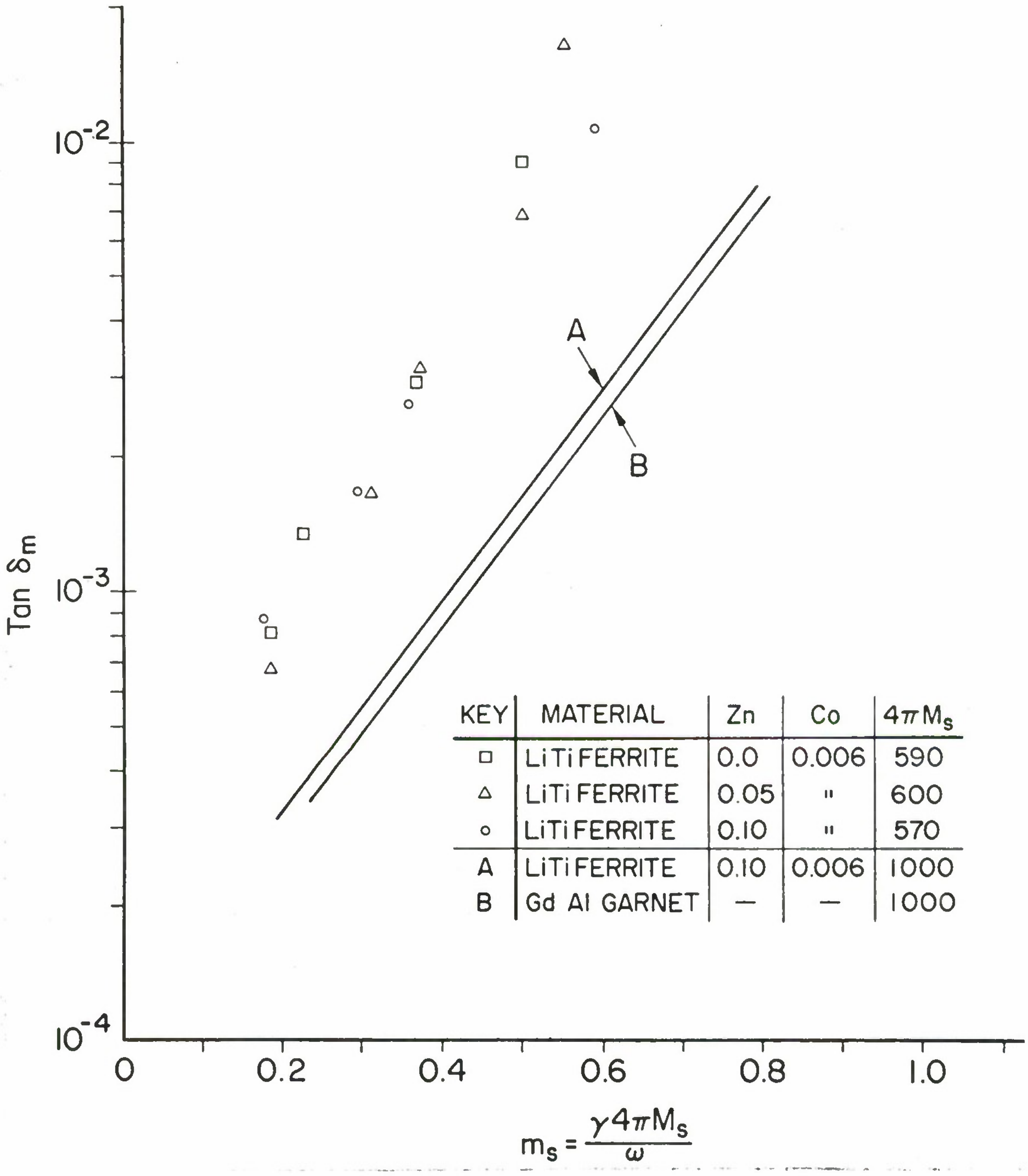

Fig. 12 Tan δ_m versus γ $(4\pi M_s)/\omega$ for several S-band lithium ferrites, a C-band lithium ferrite, and a C-band garnet.

6.0 CONCLUSION

It has been established that chemically altered dense lithium ferrites have useful microwave properties. A most important step in obtaining dense materials at low sintering temperatures was achieved by small additions of bismuth. Wide variations in properties are obtainable. Often tradeoffs among H_c, T_c, R, S, ΔH_k, tan δ_m, and tan δ_e must be evaluated for any particular application. For devices in which ΔH is important, values down to 45 Oe have been demonstrated.

At C-band and above, the properties of lithium ferrites are competitive with or superior to those of the garnets. Generally, the cost and the better temperature stability favors the former. At this point in time, the magnetic loss tangents of the S-band lithium ferrites are higher than those of the garnet counterparts. If the cost of generating the extra RF power to overcome the higher microwave losses becomes greater than the savings in materials cost, the garnets are preferred. On the other hand, the higher temperature stability of the high anisotropy compositions may be more important than the presently higher loss characteristics. It is expected that further improvements in lithium ferrite material fabrication will further extend their range of applicability.

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DOCUMENT CONTROL DATA - R&D

(Security classification of title, body of abstract and indexing annotation must be entered when the overall report is classified)

1. ORIGINATING ACTIVITY *(Corporate author)*
Ampex Corporation, Redwood City, California, under Purchase Order No. B-130 to M.I.T. Lincoln Laboratory

2a. REPORT SECURITY CLASSIFICATION
Unclassified

2b. GROUP None

3. REPORT TITLE
Development of Lithium Ferrites for Array Radar Phasers

4. DESCRIPTIVE NOTES *(Type of report and inclusive dates)*
Final Report (1 July 1969 to 31 August 1971)

5. AUTHOR(S) *(Last name, first name, initial)*
Baba, Paul D. and Argentina, Gil M.

6. REPORT DATE
30 August 1971

7a. TOTAL NO. OF PAGES
50

7b. NO. OF REFS
42

8a. CONTRACT OR GRANT NO. F19628-70-C-0230

b. PROJECT NO. 7X263304D215

c. Purchase Order No. B-130

d.

9a. ORIGINATOR'S REPORT NUMBER(S)
RR 71-13

9b. OTHER REPORT NO(S) *(Any other numbers that may be assigned this report)*
ESD-TR-71-267

10. AVAILABILITY/LIMITATION NOTICES
Approved for public release; distribution unlimited.

11. SUPPLEMENTARY NOTES
None

12. SPONSORING MILITARY ACTIVITY
Department of the Army

13. ABSTRACT

Lithium ferrites are low cost materials which are attractive for microwave device applications. For a number of years these materials have dominated the computer core industry because of excellent temperature performance and squareness of hysteresis loops. Previous attempts at exploiting lithium ferrites for microwave applications have met with limited success. Properties which required further improvements were the dielectric and magnetic loss tangents, anisotropy, coercive force, and density. This report discusses the development of microwave S- and C-band lithium ferrites with substantially improved properties, comparable to those of the more expensive ferrimagnetic garnets currently in use. Although the development of these materials has been directed at applications for latching microwave devices, they are also useful for other microwave devices.

The requirements for ferrite materials in latching phasers are reviewed, the compositional factors which result in useful properties are discussed, and performance in circulators and latching phasers is reported.

14. KEY WORDS

Lithium Titanium Ferrites
Microwave Applications
Compositional Factors
Latching Phasers
Circulators