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Exchange constant of calcium- and germanium-substituted yttrium-iron-garnet thin films

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The room-temperature exchange constant of single-crystal calcium- and germanium-substituted yttrium-iron-garnet thin films has been measured using ferromagnetic resonance techniques. The germanium concentrations varied in the range of 0 to 1.4 moles per formula unit. The experimental results are compared with the predictions of a percolation model by Kirkpatrick and Harris and reasonable agreement is found when it is assumed that the germanium substitution is confined to the tetrahedral iron sublattice. The results are also compared with the predictions of a model by Slonczewski *et al.* which relates the exchange constant in a substituted garnet to the Curie temperature. In this case the agreement is qualitative at best and raises the question of whether the exchange constant can be related to the Curie temperature without considering the details of the substitution on the individual iron sublattices. Finally the effective g value has been measured for all samples and the results are shown to be in agreement with the Wangsness model for a two-sublattice system. There is some indication of the existence of a high-field susceptibility in the heavily substituted samples.

INTRODUCTION

The Landau-Lifshitz exchange constant of calcium- and germanium-substituted yttrium iron garnet thin films has been measured using ferromagnetic-resonance techniques. Because it measures the strength of the ordering interaction in a ferrimagnetic system, the exchange constant A is useful in determining the thermodynamic properties of the material. It is of interest to determine the effect of nonmagnetic-ion substitution on A and the magnetic garnet system provides an excellent host for substitution for a number of reasons. The garnets are an insulating system in which the Heisenberg exchange interaction provides a valid description of the ordering interaction. In addition, the system allows nonmagnetic-ion substitution over a wide range of concentrations and the magnetic properties such as the Curie temperature and magnetization vary in a predictable and controllable manner. Finally, the exchange constant of the substituted iron garnets is itself of interest since these materials are presently being used in magnetic-bubble-domain devices and the exchange constant has a direct influence on many of the domain-wall properties such as the wall thickness, wall energy, and wall mobility.[1]

Previous measurements of the exchange constant of the iron garnets have relied either on techniques which are valid only at very low temperatures[2,3] or on techniques which infer the size of the exchange constant from the domain structure of the material.[4] In the latter case the results have not always been consistent because of the statistical and indirect nature of the measurements. The use of ferromagnetic resonance on thin films affords a direct measurement of the exchange constant which can, in principle, be done at all temperatures below the Curie temperature.

In this paper the results of the measurement of the room-temperature exchange constant of the system $\{YCa\}_3[Fe]_2(FeGe)_3O_{12}$ are reported. The results are compared to the predictions of a percolation theory model by Kirkpatrick and Harris.[5] Because the model is insensitive to small levels of germanium substitution on the octahedral iron sublattice, reasonable agreement is found when it is assumed that the germanium substitution is limited to the tetrahedral iron sublattice. In addition, the results are compared with a model by Slonczewski *et al.*[6] which uses the predictions of the random-phase approximation and the molecular-field theory to determine the variation of the exchange constant with the Curie temperature in the substituted garnet system.

MAGNETIC GARNETS

In the iron garnet system, there is a strong negative superexchange coupling between neighboring Fe^{3+} spins on the octahedral (a) and tetrahedral (d) sites, the spins on the two sites are aligned antiparallel and the structure has a magnetic moment equivalent to one net ferric spin per formula unit. Harris[7] has estimated the strength of the exchange interactions between the nearest neighbor sites $J_{ad} \approx -32\ \text{cm}^{-1}$ and between the next-nearest-neighbor sites $J_{dd} \approx -10\ \text{cm}^{-1}$ and $J_{aa} \approx -6\ \text{cm}^{-1}$.

Because of the different sizes and coordination of the cation sites, it is possible to substitute preferentially on either of the two iron sublattices. Geller *et al.*[8] have shown that Si^{4+} and V^{5+} substitute exclusively into the tetrahedral sublattice while Zr^{4+} and Sc^{3+} substitute exclusively into the octahedral sublattice. In addition, Ge^{4+} substitutes almost exclusively into the tetrahedral sublattice. The effect of this substitution is to lower the magnetization for tetrahedral substitution and to raise it for octahedral substitution. In both cases the Curie temperature decreases with substitution. As the level of nonmagnetic-ion substitution is increased, the effect of the two intrasublattice exchange interactions is increased relative to the nearest-neighbor intersublattice exchange interaction. This causes the spins on the unsubstituted sublattice to cant away from the aligned state with a canting angle which increases monotonically with substitution. This effect was first predicted by Yafet and Kittel[9] and discussed in detail by a number of authors.[8,10,11] This spin canting is the cause of the nonlinear variation of the magnetization of substituted garnets with composition in the high substitution range. Bozorth and Geller[12] have reported that the completely substituted garnet $Ca_3Fe_2Ge_3O_{12}$ orders antiferromagnetically at low temperatures.

EXCHANGE STIFFNESS

A number of attempts have been made to predict the dependence of the exchange stiffness constant on substitution in the substituted iron garnets. The simplest of these is derived from the molecular field theory. For the pure iron garnet such as YIG where it is assumed that there are no magnetic ions on the dodecahedral sublattice, the exchange stiffness is given by[13]

$$A = (5S/4a)(5J_{ad} - 8J_{aa} - 3J_{dd}), \tag{1}$$

where the J_{ij} represent the various exchange integrals and a is the lattice constant. From the molecular field theory of a two sublattice ferrimagnet, the Curie temperature is given by[13]

$$\begin{aligned} T_C = & -\tfrac{1}{2}(C_a N_{aa} + C_d N_{dd}) \\ & + \tfrac{1}{2}[(C_{aa} N_{aa} - C_{dd} N_{dd})^2 + 4C_a C_d N_{ad}^2]^{1/2}, \end{aligned} \tag{2}$$

where C_i is the effective Curie constant of the ith sublattice and N_{ij} is the molecular-field constant connecting the ith and jth sublattice. Under the assumption that the intersublattice exchange interactions dominate,

$$J_{ad} \gg J_{aa}, J_{dd} \quad \text{or} \quad N_{ad} \gg N_{aa}, N_{dd}, \tag{3}$$

both the exchange stiffness and the Curie temperature are proportional to J_{ad}. For a substituted material, A should be given by

$$A/A(\text{YIG}) = T_C/T_C(\text{YIG}). \tag{4}$$

This relationship neglects the effect of the intrasublattice exchange interactions which are expected to become significant as the level of substitution is increased. In addition it is expected that Eq. (4) is valid only at low temperatures because the exchange stiffness must approach zero as the measuring temperature approaches the Curie temperature.

Slonczewski *et al.*[6] have attempted to predict the temperature dependence of the exchange stiffness in the vicinity of room temperature with a model which uses the results of the random-phase approximation (RPA) and the Landau theory of second-order phase transitions. The RPA predicts the temperature dependence of A in terms of the magnetization at temperatures near T_C and the Landau theory predicts the temperature dependence of M. Combining these two predictions and normalizing to YIG with the assumption that Eq. (4) is valid at zero temperature yields the result

$$\frac{A(T, T_C)}{A_{\text{YIG}}(T, T_{C,\text{YIG}})} = \frac{T_C - T}{T_{C,(\text{YIG})} - T}. \tag{5}$$

This expression should be valid only at temperatures near the Curie temperature where the predictions of the RPA and Landau theory are valid, and it still neglects the role of the intrasublattice exchange, as did Eq. (4). Nevertheless, it does give a very reasonable looking approximation for the temperature dependence of A.

A different approach to the solution of this problem was taken by Kirkpatrick and Harris,[5] who used a percolation model. Starting with the Heisenberg Hamiltonian for an exchange coupled assembly of spins,

$$H = \sum_{ij} J_{ij} \vec{S}_i \cdot \vec{S}_j, \tag{6}$$

the spins were taken as classical vectors and the energy necessary to bend the spin system through a small angle θ over a large distance L was calculated. The equilibrium orientation of a given spin is found by minimizing the energy which gives the condition

$$\sum_j J_{ij} S^2(\theta_i - \theta_j) = 0. \tag{7}$$

The exchange stiffness is then found from the energy equation

$$E = \frac{1}{2} \sum_{ij} J_{ij} S^2(\theta_i - \theta_j)^2 = A\theta^2 L^{-2}\Omega, \tag{8}$$

where Ω is the volume. Equation (7) is equivalent to Kirchoff's current law for a network of resistors with conductances $J_{ij}S^2$ and voltages θ_i at the nodes. The energy equation, Eq. (8), then sums the power dissipation of the network, and A is proportional to the macroscopic conductivity of the network.

Kirkpatrick and Harris[5] have numerically modeled a resistor network after the garnet structure and have solved Eqs. (7) and (8) for the variation of A with nonmagnetic-ion substitution. Using the values of J_{ij} for YIG which were reported by van der Ziel *et al.*,[14] it was found that for the system

$$Y_3Fe_{3-x}Fe_{2-y}M_{x+y}O_{12}\,, \tag{9}$$

the variation of A with substitution could be described by the relation

$$A(x,y)/A(0,0)=1-0.43x-0.72y+0.25xy\,, \tag{10}$$

where x and y refer to the amount of tetrahedral and octahedral substitution, respectively. This expression is limited to compositions well above the percolation threshold, where the model indicates that the neglect of second-nearest-neighbor interactions has little effect on the results.

SPIN-WAVE DISPERSION RELATION

The exchange stiffness can be determined experimentally from the measurement of the spin-wave spectra and the magnetization. The dispersion relation for long wavelength spin waves in an infinite thin film is given by[13]

$$H=\omega/\gamma+4\pi M+\tfrac{2}{3}H_k-H_u-Dk^2 \tag{11}$$

when the external field is applied along the film normal. H is the resonance field, ω the frequency, γ the gyromagnetic ratio, $4\pi M$ the demagnetizing field, and $D=2A/M$ is the dispersion constant. H_k and H_u represent the fields due to cubic and uniaxial anisotropies.

The allowed values of k for spin waves which can be excited by a uniform rf field applied parallel to the surface of an internally homogeneous film depend on boundary conditions existing at the film surfaces. Kittel[15] initially assumed that the surface spins were completely pinned, which gave a boundary condition of the form

$$m_s=0 \tag{12}$$

at both surfaces, where m_s is the transverse component of the rf magnetization. This condition leads to a set of spin waves with k given by

$$k=n\pi/L,\quad n=1,2,3,4,\ldots, \tag{13}$$

where L is the film thickness. Spin waves with even numbered n have zero net dipole moment and will not be excited by a uniform rf field, while spin waves with odd n have intensities which fall off as $1/n^2$. Pincus[16] extended this boundary condition to the mixed form

$$\nabla m_s/m_s=a\,, \tag{14}$$

where a is the ratio of the slope of the rf component of the magnetization to the magnitude at the film surfaces. Later Soohoo[17] introduced the concept of a uniaxial anisotropy to explain the angular dependence of the surface parameter a and predicted the existence of a nonpropagating surface mode in the spin wave spectra. Finally Puszkarski[18] derived a microscopic form of the surface inhomogeneity model which has had excellent success in explaining much of the observed behavior of the spin-wave spectra. According to the mixed boundary condition expression, Eq. (14), there is a continuum of surface pinning conditions which can exist in addition to the one in Eq. (12). If there is free precession on one surface, $\nabla_n m_s=0$, and complete pinning on the other, $m_s=0$, the allowed values of k are given by

$$k=n\pi/L,\quad n=\tfrac{1}{2},\tfrac{3}{2},\tfrac{5}{2},\ldots, \tag{15}$$

and the intensities of the modes are proportional to $1/n^2$. Another boundary condition which can exist is that of free precession at both surfaces. In this case the uniform precession mode, $k=0$, is the only mode having a net dipole moment. In addition to these, many intermediate pinning conditions have been observed.[19]

COMPOSITION MEASUREMENTS

The samples used in this work were single-crystal thin films of $\{Y_{3-x}Ca_x\}[Fe_2](Fe_{3-x}Ge_x)O_{12}$ grown at Bell Laboratories by liquid phase epitaxy on [111] oriented $Gd_3Ga_5O_{12}$ substrates. The thickness was in all cases approximately 1 μm and compositions were in the range $0<x<1.4$. The compositions were estimated by measuring the depression of the Curie temperature from that of pure YIG (553 K) and comparing with the data on polycrystalline samples by Winkler *et al.*[20] These data are plotted in Fig. 1, where the solid line represents the polycrystalline sample data and the points represent the samples used in this work. It has been found that the Curie temperature of an LPE (liquid-phase-epitaxy) film is inevitably depressed by lead contamination from the growth flux. However, as calcium is added to the film the amount of this lead contamination decreases. It was assumed that for films with Curie temperatures less than 530 K, the lead contamination was negligible and the compositions of these films were estimated by a direct comparison to Winkler's data. To estimate the composition of the samples with Curie temperatures greater than 530 K, the graph was extrapolated to give a T_C at 543K for YIG. This was a typi-

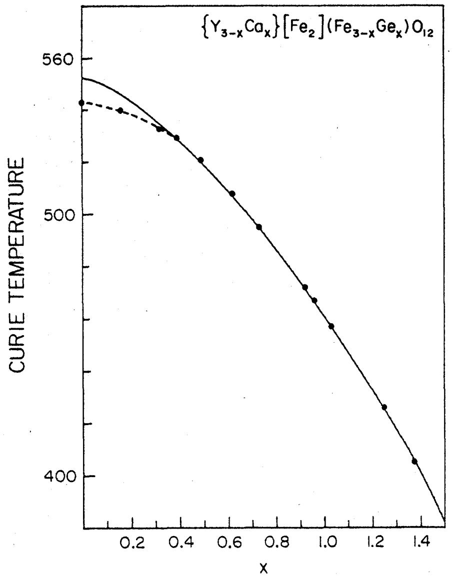

FIG. 1. Variation of the Curie temperature with composition in $\{YCa\}_3[Fe]_2(FeGe)_3O_{12}$ (after Winkler *et al.*) (Ref. 20).

cal value for the Curie temperature of a YIG film grown from the melt before the addition of calcium and germanium.

MAGNETIC MEASUREMENTS

A typical room-temperature spin-wave spectrum of a YIG:CaGe film is shown in Fig. 2. This spectrum was measured at 9.5 GHz with the applied field along the film normal. The large number of spin-wave modes detected indicates that the surface pinning is quite strong ($m_s \approx 0$ at the surfaces), while the fact that even-numbered modes are detected indicates that there is some asymmetry in the boundary conditions and that the spectrum corresponds to an intermediate case between those described in Eqs. (13) and (15).

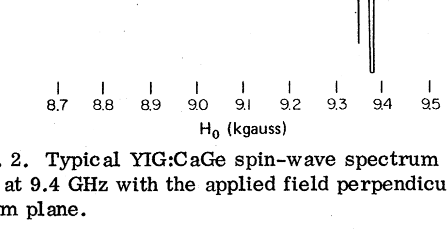

FIG. 2. Typical YIG:CaGe spin-wave spectrum measured at 9.4 GHz with the applied field perpendicular to the film plane.

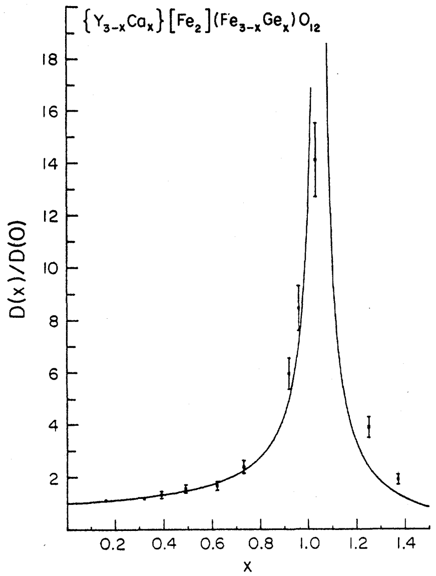

FIG. 3. Variation of the dispersion constant with composition in YIG:CaGe.

The customary method of determining the dispersion constant from the spin-wave spectrum is to assume a form of the boundary conditions given either by Eq. (13) or Eq. (15) and plot the resonance field for the different modes against n^2. If the first few modes are neglected this plot yields a straight line with slope proportional to D. However, experimentally it is often difficult to determine which of these two boundary conditions is closest to that actually existing in the film. The assumption of either of these two boundary conditions can lead to quite sizable differences in the value of D which is determined. If the field separation between adjacent modes is plotted against the difference in the squares of the mode numbers, the slope of this plot is proportional to the dispersion constant for a wide range of boundary conditions. Detailed analysis shows that the error is less than 3% over the entire range of physically reasonable boundary conditions.

In Fig. 3 the variation of the dispersion constant with composition is plotted. These results are all taken from room-temperature data measured at 9.5 GHz and the dispersion constant values are normalized to the bulk value for pure YIG which is taken to be 5.16×10^{-9} Oe cm^2.[21] The error bars represent the uncertainty in the film thickness which is taken to be 5% in all cases. The solid line is obtained by using the predictions of the Kirkpatrick and Harris[5] model for the dependence of

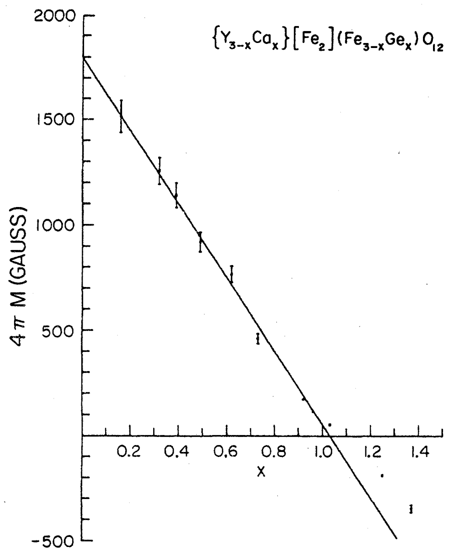

FIG. 4. Variation of the room temperature magnetization with composition in YIG:CaGe.

the exchange stiffness A and by assuming a linear variation of the magnetization with composition is due to this compensation in M. The linewidths of all samples except $x=1.03$ were less than 4 G. The $x=1.03$ sample is near the compensation point and has a large uncertainty due to its large linewidth which was about 200 G. It is suspected that this increase in linewidth is due to inhomogeneities in the film. As a result, only four spin-wave modes were detected in this sample and an accurate determination of D quite difficult to obtain. The last two points fall above the curve because the magnetization dependence departs from linearity in this region and the exact location of the compensation point is unknown. From this graph it is difficult to draw many conclusions about the behavior of the exchange stiffness since the main influence comes from the variation of M.

The room-temperature magnetization of all samples was measured using a vibrating sample magnetometer and results are plotted in Fig. 4. The error bars again represent the uncertainty in the film thickness, not the total uncertainty, which is expected to be similar in all cases except for the sample at $x=1.03$. This sample is very near the compensation point and its moment is approaching the limit of sensitivity of the magnetometer. The negative moment of the last two points indicates that these samples are beyond the compensation point and have moments dominated by the octahedral sublattice. For complete tetrahedral substitution it would be expected that the compensation point be at a composition near $x=1$. Geller *et al.*[8] have made low-temperature magnetization measurements on the YIG:CaGe system and report the compensation point to be at $x=1.03$ at 0 K. The direction of the Faraday rotation of the sample $x=1.03$ indicates that it has a moment which is dominated by the tetrahedral sublattice. It is estimated that the compensation point is shifted upward slightly because the measurements were made at room temperature.

As can be seen from the graph, the magnetization does decrease in a nearly linear manner with substitution up to about $x=1$. The solid line in the figure is the best straight-line fit to the data with the last two points deleted from the analysis. The departure of these two points from the straight line is probably an indication of spin canting on the octahedral sublattice. Previous measurements[8,20] on the YIG:CaGe system show this same departure from linearity in this range of compositions.

Upon combining the measurements of the magnetization and the dispersion constant, a value of the exchange stiffness $A=\frac{1}{2}MD$ is obtained. The results of this measurement are plotted in Fig. 5. The solid line is the best straightline fit to the data with the sample at $x=1.03$ excluded from the analysis. The value of the room-temperature exchange stiffness of YIG is taken to be $A=3.75\times 10^{-7}$ erg/cm, which is well within the range of previously reported values.[21,22] The equation of this line is given by

$$A(x)/A(0)=1-(0.48\pm 0.03)x\,. \tag{16}$$

It is interesting to compare this result with the prediction of the Kirkpatrick and Harris[5] model, which says that for substitution exclusively on the tetrahedral sublattice the variation should be

$$A(x)/A(0)=1-0.43x\,. \tag{17}$$

The small difference between the two expressions cannot be explained on the basis of a partial octahedral substitution as the model is insensitive to small changes in the relative substitution between the two sites. Using Eq. (10), it would require al-

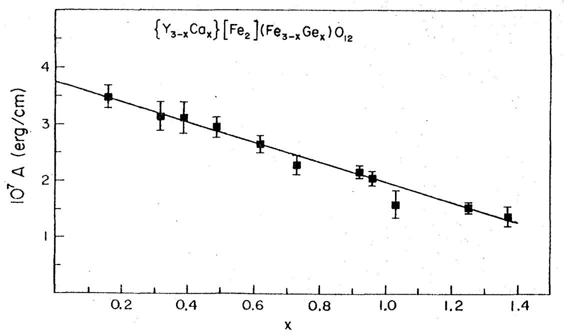

FIG. 5. Variation of the exchange constant with composition in YIG:CaGe.

most 20% substitution on the octahedral sublattice to explain the difference. That large a substitution on the *a* sites would have a very significant effect on the slope of the composition dependence of the magnetization and on the location of the compensation point.[8] A more likely explanation is that room temperature data is compared with a low-temperature model. The room-temperature A should fall more rapidly with substitution than the low-temperature A since the room-temperature A must go to zero as the Curie temperature approaches room temperature. Equation (16) can be extrapolated to predict that A vanishes at a composition just above $x = 2.0$, while the Curie temperature reaches room temperature at a composition of approximately $x = 1.9$.

A comparison can also be made between the model prediction of Slonczewski *et al.*[6] and the data measured here. This is done in Fig. 6, where the solid line is the model prediction. As can be seen, the agreement is qualitative at best and it improves as the Curie temperature approaches room temperature, where the approximations in the model are most valid. This same general behavior has been seen in measurements[22] of $(YLA)_3(GaFe)_5O_{12}$.

It is valid to again raise the question as to whether it is valid to try to relate A to T_C without considering in detail the degree of substitution on each of the two iron sublattices. If it is assumed that the model prediction of Eq. (10) is at least approximately correct and if the results of Winkler *et al.*[20] are used to relate the Curie temperature to the composition for both octahedral and tetrahedral substitution, an empirical relationship between A and T_C can be obtained. This relationship differs significantly for octahedral and tetrahedral substitution. The curve for tetrahedral substitution is concave upward as is the data in Fig. 6, while the curve for octahedral substitution is nearly linear and there are significant deviations between the two curves. Plans are presently being made to quantitatively test this hypothesis on a system with octahedral substitution.

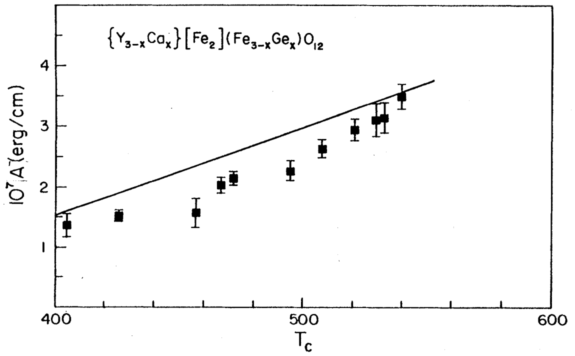

FIG. 6. Variation of the exchange constant with Curie temperature in YIG:CaGe. The straight line is the prediction of Slonczewski *et al.* (Ref. 6).

FREQUENCY DEPENDENCE

The dispersion constant has been measured on all samples at 9.5 and 23 GHz and for a few samples at 34 GHz. It has been found in all cases that the measured value of D decreased with increasing frequency. The change in the measured value of D was as high as 15% in some samples. Unfortunately there was no correlation between the rate of decrease of D with frequency and the film composition, and it is difficult to explain the cause of this phenomenon. A partial explanation is given by the increase of the resonance linewidth with increasing frequency. Because of this, fewer spin-wave modes can be detected at the higher frequencies and the uncertainty in the measured value of D is increased. This would be expected to give random changes in the measured result. Another possible explanation is the existence of a high-field susceptibility in the spin system. Anderson[23] has measured the temperature dependence of the susceptibility of YIG below the Curie temperature, and at room temperature found the result

$$\chi \approx 0.02 \text{ emu/g KOe} . \qquad (18)$$

An increase of the applied field by 5 kOe, which is the approximate difference between the resonance field between 9.5 and 23 GHz, will cause an increase in M of 0.3% and a decrease in D of the same amount. For substituted YIG the susceptibility should be larger since the ratio of the applied field to the exchange field is larger and since the measurement temperature is closer to the Curie temperature, where χ diverges. However, from this data it is difficult to estimate just how large this effect should be.

EFFECTIVE *g* VALUE

In addition to the measurement of the dispersion constant, the effective g value of all samples has been measured from the frequency dependence of the resonance field. The results are plotted in Fig. 7. It is seen that g_{eff} diverges at a point near $x = 1$. This result is in agreement with the Wangsness model[24] for a two-sublattice system where

$$g_{\text{eff}} = \frac{M_1 - M_2}{M_1/g_1 - M_2/g_2} . \qquad (19)$$

$M_{1,2}$ and $g_{1,2}$ are the magnetization and g values of the individual sublattices. The location of the point at $x = 1.03$ may be explained as follows. The experiment does not measure g but its absolute value, and this sample has a composition just beyond the region at which [illegible]es. It can also be seen

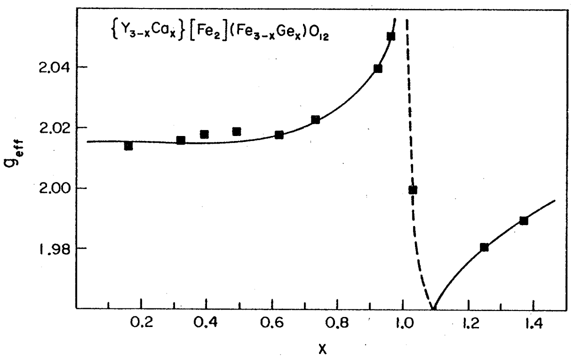

FIG. 7. Variation of the effective g value with composition YIG:CaGe.

that the curve rises less rapidly after the compensation point than before it. This effect is also consistent with the existence of a high-field susceptibility. If the magnetization is given by

$$M = M_0 + \chi H , \tag{20}$$

the resonance equation, Eq. (11), neglecting the spin-wave energy and anisotropy terms, becomes

$$\omega = \gamma(1 - 4\pi\chi)H - 4\pi\gamma M . \tag{21}$$

The measured value of γ or g is then reduced by a factor of $1 - 4\pi\chi$. The size of this effect can be estimated by calculating the susceptibility necessary to lower the g value of the last sample from 2.02 to its measured value of 1.99. The value of χ necessary to accomplish this is $\chi = 0.2$ emu/g kOe, which is only a factor of 10 greater than the measured room-temperature susceptibility of YIG.[23] It is reasonable to suspect that the shape of the curve of g_{eff} is due at least in part to this effect.

CONCLUSIONS

The room-temperature exchange stiffness constant of $\{YCa\}_3[Fe]_2(FeGe)_3O_{12}$ has been measured over a broad range of compositions, and the results have been compared with the predictions of a percolation model by Kirkpatrick and Harris. Considering that the model is a low-temperature model, the agreement is quite reasonable. The results have also been compared to the predictions of a model by Slonczewski *et al.*, which relates the strength of the exchange constant at room temperature of the Curie temperature. In this case the agreement is qualitative at best and raises the question as to whether a relationship can be made between A and T_C without first considering the details of the substitution on each of the magnetic iron sublattices.

Finally the effective g value of all samples has been measured and is shown to agree with the Wangsness relation for a two-sublattice ferrimagnet. In addition, the existence of the high-field susceptibility in the more heavily substituted films is indicated.

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