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REVERSIBLE PROPERTIES OF POLYCRYSTALLINE FERROMAGNETS

II. EXPERIMENTAL VARIATION OF THE REVERSIBLE SUSCEPTIBILITY AND Q WITH MAGNETIZATION

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Solid State Devices Laboratory
Department of Electrical Engineering

By: D. M. Grimes

Approved by: H W Welch Jr.
H. W. Welch, Jr.

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INTRODUCTION

The reversible susceptibility and the differential magnetostriction of ferromagnetic and ferrimagnetic material has been considered from a statistical standpoint in a previous paper,[1] which will be designated I wherever referred to in this present paper. The variation of both quantities with magnetization was considered for differential fields applied both parallel with and normal to the effective biasing field and for models assuming that the reversible susceptibility had its origin in domain-wall motion and in domain rotation respectively when the static moments were oriented along directions of minimum anisotropy energy.

The magnetic Q is considered to be the ratio of the real to imaginary part of the reversible susceptibility when that susceptibility is measured at a finite frequency. The present paper considers the variation with magnetization of Q for biasing and differential fields parallel and normal respectively.

Detailed measurements are shown for four different ferrite materials.

THE MAGNETIC Q.

REVERSIBLE SUSCEPTIBILITY BY DOMAIN ROTATION. The variation of the magnetic Q with magnetization and effective biasing field can be seen from Eq. 9 of I. Substituting the values of χ_+ and χ_-, the susceptibilities of the positively and negatively rotating field, from Eq. 5 of I into Eq. 9 of I, the resultant magnetic Q can be written as

$$Q = \frac{\omega_1}{\omega\epsilon} (1 + \epsilon^2) \left\{ \frac{1 - \left(\frac{\omega}{\omega_1}\right)^2 \frac{1}{1 + \epsilon^2} \left(\frac{1 - \epsilon^2}{1 + \epsilon^2}\right)}{1 + \left(\frac{\omega}{\omega_1}\right)^2 \frac{1}{1 + \epsilon^2}} \right\}, \tag{1}$$

for both parallel and transverse fields, and for values of applied angular frequency small compared to ω_1,

$$Q = \frac{\omega_1}{\omega\epsilon} (1 + \epsilon^2). \tag{2}$$

The symbols are as defined in I, where ω is the radial frequency, $\omega_1 = \gamma H_{an}$ with H_{an} the effective anisotropy field and γ the magnetomechanical ratio, and ϵ is a constant proportional to the loss term of the differential equation describing the motion of the magnetic moments. From Eq. 7 of I, ω_1 of Eq. 1 can be replaced by $\omega_1 + \rho\omega_o = \gamma M_s$, where ρ is described in I. From the form of Eq. 2 it is apparent that any variation of Q with magnetization is dependent upon the ρ and ϵ variation. The applied field H_{ap} acts as an addition to the effective anisotropy field. The parameters ϵ and ρ must be, in general, allowed to vary with both H and M. ϵ is dependent upon the intrinsic nature of the material and, as such, is relatively insensitive to the magnetization level. ρ is nearly constant. In the low frequency limit, the initial susceptibility $\chi_o = \frac{1}{3} (\chi_- + \chi_\chi)$ is given by: (see Eqs. 5 and 10 of I)

$$\chi_o = \frac{2\omega_o}{3\omega_1} \quad , \tag{3}$$

so the product $\chi_o Q$ can be written as:

$$\chi_o Q = \frac{2\omega_o}{3\omega\epsilon} (1 + \epsilon^2) \tag{4}$$

This expression depends upon H_{ap} and M only through the ϵ variation, which must surely be slowly varying for at least low magnetic fields. Combining Eq. 4 with E.. 9 of I:

$$Q\chi^r_{rt} = \frac{\omega_o}{2\omega\epsilon} (1 + \epsilon^2)(1 + <\cos\theta>); \; Q\chi^r_{rp} = \frac{\omega_o}{\omega\epsilon} (1 + \epsilon^2)(1 - <\cos^2\theta>). \tag{5}$$

Eq. 5 should be valid to larger values of M than Eq. 9 of I and would be limited by the static moments remaining aligned along crystallographic easy directions. According to Amar[2] this is valid to higher field strengths in polycrystalline material than would be expected from the single crystal parameters.

For domain rotation the Q will thus be the same monotonic increasing function for parallel and transverse fields.

REVERSIBLE SUSCEPTIBILITY BY WALL MOTION. Eq. 21 of I is the differential equation of motion utilized to described the motion of a domain wall. The change in magnetic moment due to the movement of 180° walls is given by:

$$\Delta M = \frac{1}{V} \sum_i 2M_s A_i x_i \tag{6}$$

where A_i is the area of the i'th wall and x_i is the distance through which it is moved under the influence of the applied magnetic field ΔH and V is the volume of material. From Eq. 6 and Eq. 21 of I it follows that

$$\chi_r^w = \frac{1}{V}\sum_i \frac{2M_s A_i}{\alpha_i}\left\{\frac{1 - \omega^2\left(\frac{m}{\alpha_i}\right)}{1 + \omega^2\left[\left(\frac{\beta}{\alpha_i}^{2} - \frac{m}{\alpha_i}\right)\right] + \omega^2\left(\frac{\omega}{\alpha_i}\right)^2}\right\} \tag{7}$$

and that the magnetic Q is given by:

$$Q = \frac{\sum_i \frac{A_i}{\alpha_i}\left(1 - \omega^2 \frac{m}{\alpha_i}\right)}{\sum_i A_i/(\alpha_i)^2\, \omega\beta} \; . \tag{8}$$

The low frequency limits are

$$\chi = 2M_s \sum A_i/\alpha_i, \quad \text{and } Q = \frac{1}{\omega\beta}\,\frac{\sum A_i/\alpha_i}{\sum A_i(\alpha_i)^2} \; . \tag{9}$$

The constant β is related to ϵ through a series of magnetization and field independent parameters.[3] The remaining variable portions of Eq. 9 are the terms involving A_i and α_i. The expected variation of $\sum_i A_i/\alpha_i$ can be seen fron Eqs. 2 and 18 of I. α_i would presumably increase with moment for the parallel fields, so the Q would also increase. For the case of transverse magnetic fields the case is not so clear cut although from Eq. 18 of I, A_i/α_i must decrease with increasing field. The variation of the α_i would presumably depend on the origin of the restoring force and for large applied field α_i would increase with H_{ap}. However, there is no obvious reason to expect the α_i to increase and indeed they might decrease with H_{ap} for small values of H_{ap}. It is thus to be expected that the magnetic Q for wall-motional susceptibility will increase with M for parallel fields but increase more slowly or even decrease with M for low M until finally increasing with M for high M in transverse fields.

HYSTERESIS EFFECTS

Remaining hysteresis effects in the susceptibility versus magnetization plots, when the susceptibility is due to domain-wall motion, has been discussed[4] in terms of the metastable volume of material with moment remaining oriented in the direction of the previous saturation magnetization. This metastable volume is considered to contribute a net magnetization in the direction of the previous saturation, but to contribute nothing to the parallel field susceptibility. Thus, the susceptibility goes through a peak value always less than the susceptibility of the virgin material at a value of magnetization finite and in the direction of the previous saturation field.

If the gross magnetization is presumed to change by rotation of the domain moments, then after the material has been saturated as the field is decreased the moments again rotate in easy directions. If, as the saturating field is decreased, the domain moments rotate until the component perpendicular to the field is the same as in the virgin material, but all components originally parallel and anti-parallel with the field remain parallel, then the value of remanent magnetization will be $0.5M_s$ and the susceptibility due to domain rotation, and thus proportional to $[1 - \langle\cos^2\theta\rangle]$, will, at this point, just equal that of virgin material. This is the model used by Fomenko[5] to interpret his results. If this is also the position of the maximum number of atomic moments normal to the field direction, then it will be, in turn, the position of maximum parallel and minimum transverse field susceptibility. For parallel fields, then, the maximum would occur for finite decreasing M, but would equal in value the susceptibility of virgin material.

If the hysteresis action is such as to increase the parallel-antiparallel component, then χ_{rt} would always exceed χ_{rp}. Reviewing, in both the rotational and wall-motional models the peak parallel susceptibility would occur for a decreasing magnetization, but the ratio of peak to initial should be less for rotation than for domain wall motion. These results are based upon the assumption that in static fields all moments are in crystallographically "easy" directions. Thus the changes in finite fields are considered to be either wall motional or to be rotational between easy axes. That there is often a difference between the type of processes depending upon the size of signal has been demonstrated.[6]

SAMPLES. To test the hypothesis of I regarding the variation of the susceptibility and of the previous section regarding the magnetic Q, it was decided to measure a series of ferrites with widely varying anisotropic and magnetostrictive constants. Now the room temperature saturation magnetization is positive for magnetite and negative for cobale and nickel ferrite.[7] The first order anisotropy constant of cobalt ferrite is positive, that of nickel and iron ferrite is negative.[7] Small zinc ferrite additions increase the susceptibility of each of the above ferrites. These constants effect the susceptibility in the manner to be described.

The initial susceptibility due to wall motion has been approximated by Becker[8] for sinusoidal internal strains arising from magnetostrictive forces which arose when the material was cooled through its Curie temperature. The result is

$$\chi_o = \frac{8\pi M_s^2}{9\lambda_s^2 E} \tag{10}$$

where λ_s is the saturation magnetostriction and E is the Young's modulus of the material. Likewise the initial susceptibility due to domain rotation can be approximated by[9]

$$\chi_o = \frac{2\mu_o M_s^2}{3K_1} , \tag{11}$$

where μ_o is the permeability of free space and K_1 is the first order anisotropy constant.

In an attempt to vary the ratio of wall motional to domain rotational susceptibility the ratio of nickel and cobalt was varied in mixed iron-cobalt-nickel-zinc ferrites. To test the equations of I describing the effect of a biasing magnetization on the domain rotational transverse susceptibility, it is necessary to have the anisotropy sufficiently high to assure that the moments are aligned along the easy crystallographic directions and still small enough to allow a measurable susceptibility due to the rotation against the effective anisotropy fields. To accomplish the manufacture of such ferrites, a series of fifteen compositions (shown in Table I) with different amounts of iron, nickel and cobalt were manufactured under the following conditions: The material was weighed out, ball milled in a steel mill with a thin acetone slurry for six hours, pressed into a toroidal shape of one inch O.D., 5/8 inch I.D. and about 1/4 inch thick, placed on an alundum tray which had been rubbed with Fe_2O_3, and heated rapidly to 1150°C. They were then heated at about 60°C/hr. to 1375°C, held for 1/2 hour, then the temperature reduced to 1200° and held for two hours. They were then slowly cooled to 1100°, flushed with nitrogen and furnace cooled in a nitrogen atmosphere. The resultant ferrites were black and of about 0.95 X-ray density. They were toroidally wound, and then placed between the pole faces of a battery powered electromagnet. The pole faces were tightened snugly to an insulating sheath over the windings and the toroidal windings taken to a Boonton Type 260-A Q-Meter. For a preliminary survey, the capacitances and Q were measured at 500 kc/sec as a function of the magnet currents. From these fifteen specimens, F-1-2, F-6-2 and F-10-2 were chosen for detailed measurements. In addition, a sample of magnesium ferrite designated I-15-1 was measured in detail. Magnesium ferrite was chosen as a test case since it was in this material that Rado obtained striking evidence of the existence of both wall motional and domain rotational susceptibility resonances.[10]

TABLE I

COMPOSITION OF THE FERRITES SURVEYED

	Moles of oxide added to mix.*			
Designation	NiO	CoO	ZnO	Fe_2O_3
F-1	.2337	0	.2263	.5000
F-2	.2314	.0023	"	"
F-3	.2290	.0047	"	"
F-4	.2150	.0187	"	"
F-5	.1963	.0374	"	"
F-6	.0843	.1496	"	"
F-7	.2065	0	.2294	.5641
F-8	.2044	.0021	"	"
F-9	.2024	.0041	"	"
F-10	.1900	.0165	"	"
F-11	.1735	.0330	"	"
F-13	.0743	.1322	"	"
F-14	.1613	0	.1791	.6596
F-15	.1484	.0129	"	"
F-16	.1355	.0258	"	"

*The amounts are listed for CoO for comparison, an equivalent amount of Co_2O_3 was added. All oxides were commercially available C. P. grades.

EXPERIMENTAL TECHNIQUE: The experimental technique utilized to determine the susceptibility and the magnetic Q is the same as that described earlier,[4] except the high frequency measurements were taken with the toroidal leads connected to a Q-meter instead of a VTVM. The frequencies used were 320 kc/sec and 500 kc/sec. The lower frequency was used for all except F-1-2, to avoid insofar as possible transverse field magnetostrictive resonances. For a check on the susceptibility variation, certain data were repeated at 10 kc/sec using the previous technique.

RESULTS: The results of the measurements are presented in Figs. 1 through 4. Plots of the normalized susceptibility and the Q are shown as the ordinates with the magnetization as abscissa. The direction of change of magnetization is indicated by the arrow. Figs. 5 through 8 show the symmetric and antisymmetric parts of the susceptibility and the susceptibility-Q product as a function of magnetization. Table II shows the measured values of Q and susceptibility at zero magnetization, the saturation magnetization, the remanent magnetization, and the coercive force.

TABLE II

PARAMETERS OF THE MEASURED FERRITES

				Values for M = 0 320 kc/sec			
				Parallel		Transverse	
Specimen	H_c $\frac{amp}{m}$	M_s $\frac{amp}{m}$ x 10^{-5}	M_r $\frac{amp}{m}$ x 10^{4}	Q_o	χ_o	Q_o	χ_o
F-6-2	125	2.79	1.84	72.4	46.5	60.8	52.8 49.6+
F-1-2	20.3	3.10	1.89	13.2	388*	13.5	450*
F-10-2	193	2.38	1.21	71.5	153	68.8	163
I-15-1	175	1.23	0.98	34.6	51.2 42.4+	38.8	54.2 45.4+

* f = 500 kc/sec

\+ f = 10 kc/sec

F-6-2. Data from specimen F-6-2 are shown in Figs. 1 and 5. The transverse susceptibility fits the expected curve for domain rotation to greater than 0.6 M_s. This fact and the nearly constant Q leads to the conclusion that if only wall motional and domain rotational processes exist then the susceptibility in low fields must be due almost exclusively to domain rotation and $H_{an} \gg H_{ap}$ for at least $M \leq .5\ M_s$. The slight decrease in Q with magnetization for transverse fields is believed due to magnetostrictive losses still present at 320 kc/sec. The parallel field susceptibility peaks at about a decreasing 0.3 M_s. The lack of symmetry in the parallel case is difficult to interpret in view of Eq. 10 of I and the symmetry of the transverse field data. Fig. 9 compares the experimental parallel field susceptibility with that calculated from the experimental data for transverse fields using Eq. 10 of I. Some possible sources of the lack of symmetry are (1) that the domain structure may be different for magnetizations around the toroid and parallel to its axis, so a real difference in the hysteresis may exist in the two cases. Thus some wall motional susceptibility may exist in parallel fields, not present in transverse fields. (2) It is also possible that the moments are not located in easy crystallographic directions, so the restoring force is asymmetric. (3) The fact that the applied alternating signal was of finite magnitude might effect the two cases differently. Simultaneous differential measurements of parallel and transverse susceptibilities on a sphere would seem to be in order.

The relaxation frequency due to domain rotation can be written as

$$f = \frac{\gamma}{2\pi} \frac{2M_s}{3X_o} . \tag{12}$$

Any sizeable contribution to the susceptibility from domain wall motion should invalidate an experimental check of Eq. 12. The frequency calculated for F-6-2 is found to be about 150 mc/sec. The spectrum of a specimen of this material was measured and found to have but one resonance which occurred at about 125 mc/sec.[11]

F-1-2. Data for specimen F-1-2 are shown in Figs. 2 and 6. Since this specimen is a nickel-zinc ferrite, it was expected that the anisotropy energy would be smaller than for F-6-2,[7] so rotational susceptibility was expected. From Figs. 3 and 10 it is apparent that the susceptibility does not follow the predicted curve for domain rotation based upon the static moments being oriented along easy crystallographic directions. However, the curve XQ does follow the expected curve to about M = 0.5 M_s, and, further, the magnetic Q increases monotonically from a broad minimum value at about $M = 0.2\ M_s$ (decreasing), as does the Q for parallel fields. This behavior is consistent with that expected of domain rotation (for a small value of H_{an}). It is therefore concluded that the susceptibility is probably due to domain rotation, but the effective anisotropy fields are not sufficiently large to maintain the static moments along easy crystallographic directions. The anisotropy field can be estimated as that field for which the Q is twice its initial value. Thus the averaging equations for the susceptibility as derived in I can only be expected to be valid to relatively small values of M/M_s. The parallel field data shown in Fig. 4 is consistent with the above description, as is the large ratio of peak Q to that near $M = 0$. This material is found to have but a single relaxation at about 35 mc/sec.[11]

F-10-2. Data for specimen F-10-2 are shown in Figs. 3 and 7. The striking features of this material are that both susceptibilities go through a single maximum, and that both maxima are displaced to the left of center by about $0.3\ M_s$. The Q increases with field more rapidly with parallel than transverse fields, and the shape of the susceptibility curves as well as the susceptibility-Q curves are indicative that the mechanism of change in gross and reversible moments is the same. The Q variation indicates that the reversible susceptibility is due to wall motion. The material exhibits a striking drift in the susceptibility and Q after the biasing field has been changed, for magnetizations up to about $0.7\ M_s$. For lower magnetizations it was necessary to delay all readings by a minimum of two minutes after altering the field. This drift is indicative of changes in the gross moment by wall motion, a fact not inconsistent with the results of Epstein.[12] The initial susceptibility frequency spectra shows a pronounced peak at about 15 mc/sec and a resonant frequency of about 50 mc/sec,[11] but no second peak is observed. If the susceptibility is due to wall motion, then the rotational contribution must be completely negligible. This implies that the anisotropy constant

K_1 must be very large and the magnetostrictive constant very small. It is concluded that the wall motional concept is the only concept consistent with all the observed facts. This is not inconsistent with the data of reference 7. Fig. 10 compares the parallel susceptibility calculated from the transverse field data using Eq. 20 of I in the form:

$$\chi^w_{rp}/\chi_o = \frac{\chi^w_{rt}/\chi_o}{1 - \dfrac{d(\ln\chi^w_{rt}/\chi_o)}{d(\ln M/M_s)}} \tag{13}$$

with experimental values.

I-15-1. The magnesium ferrite I-15-1 is of the same material reported on by Rado, Folen and Emerson.[13] The transverse field magnetic Q increases with applied field, but less rapidly than does the parallel field Q. The susceptibilities both have but a single maxima, but χ_{rt} Q vs M shows the double peak characteristic of rotation. The symmetrized values of the transverse field variables lie between that expected by domain rotation and by wall motion. Thus it is concluded that both mechanisms are of importance in this sample, in agreement with conclusions based upon the frequency spectrum.[13]

DISCUSSION.

An analysis of the relationships between the remanant condition and the reversible susceptibility has been carried out by Frei and Shtrikman[14] on the assumption that the reversible susceptibility is due to domain rotation. One item they did not point out is that their analysis assumes not only that the reversible quantities be due to domain rotation but also the expansion of magneti-

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[*] Frei and Shtrikman independently derived Eq. 10 of I at the remanant point.

zation in terms of applied field must obey the rotational equations through at least second order in the ratio of applied to anisotropy fields. This can be seen in their Eq. 10 from which the remainder of their work is derived. The validity of the averaging equations of I for wall motions rest upon the domains remaining always oriented in easy crystallographic directions. Conversely, if the anisotropy were zero then the equations for wall motion could be immediately carried over for domain rotation. Sample F-6-2 apparently fits the conditions of I but not the conditions of Frei and Shtrikman. Sample F-1-2 did not fit the conditions of I, yet had susceptibility predominately due to domain rotation. It must therefore be expected that this sample would more nearly fit the conditions assumed by Frei and Shtrikman. This is verified in Table III. The theory would not be applicable for I-15-1 and presumably not for F-10-2. The measured ratio of remanent to saturation magnetization is compared in Table III with that calculated by Frei and Shtrikman's Eq. 29. The striking disagreement for F-6-2 and I-15-1 is also obvious.

TABLE III

COMPARISON OF MEASURED AND CALCULATED REMANANCE

	M_r/M_s	
Specimen	Measured	Calculated*
F-6-2	.66	.11
F-1-2	.61	.51
F-10-2	.51	.76
I-15-1	.80	1.53

* Calculated using Frei and Shtrikman's Eq. 29

CONCLUSIONS

The expected variation of the magnetic Q with magnetization has been discussed for the cases where the susceptibilities have their origin in domain rotation and in domain-wall motion, and the nonzero static magnetization exists with moments along easy crystallographic directions. Detailed experimental results on four samples are reported and analyzed in terms of the expected Q variation as well as the expected susceptibility variation described in I. It is concluded that two of the samples have susceptibilities arising very predominately from domain rotation, one from wall motion and one from both. It is assumed that no other mechanisms are operative. It is concluded that Frei and Shtrikman's analysis of the remanent position is valid under only the more restricting conditions described herein.

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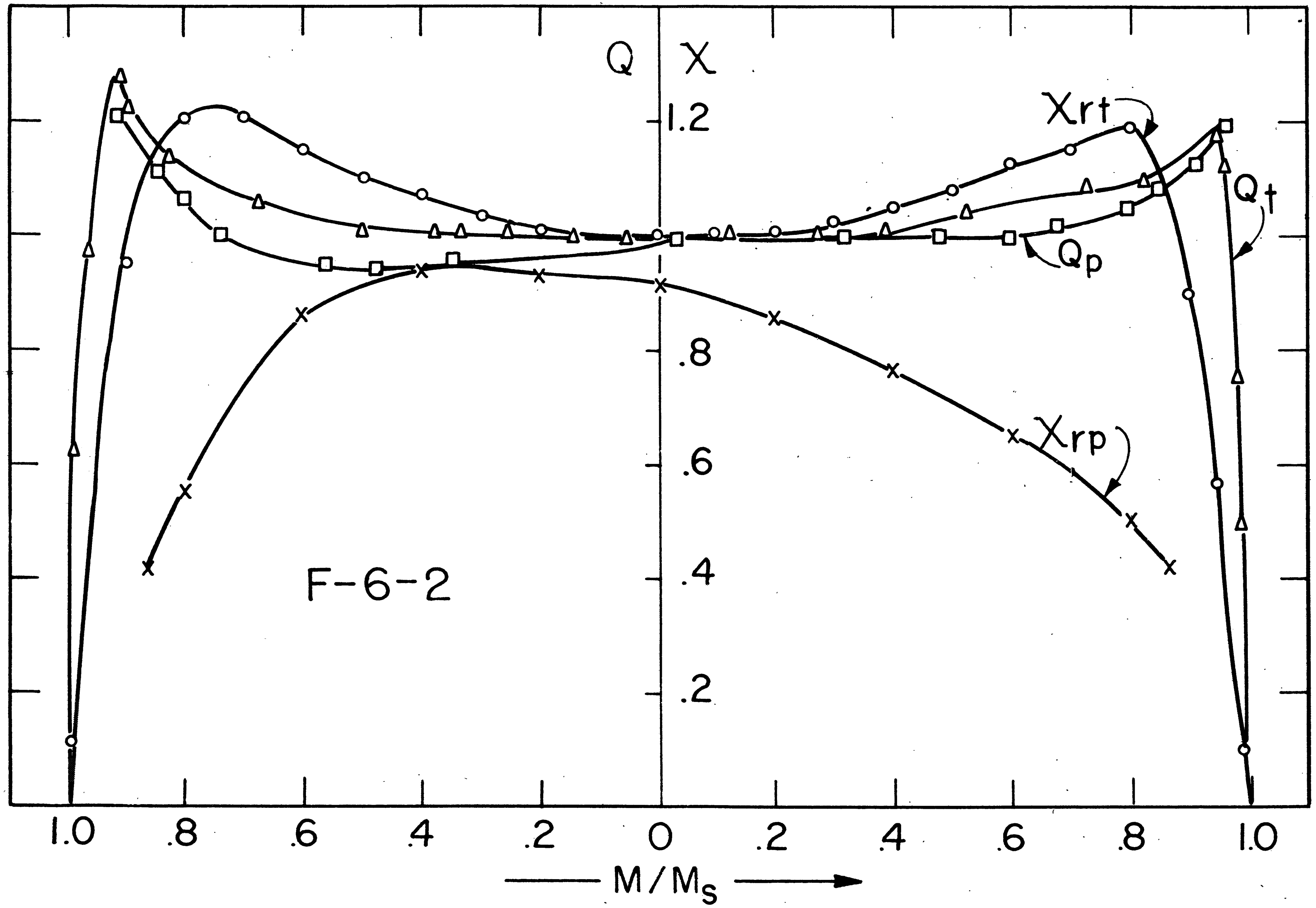

Fig. 1. The Variation of the Normalized Susceptibilities and Q with Magnetization for F-6-2. The arrow indicates the direction of change of magnetization.

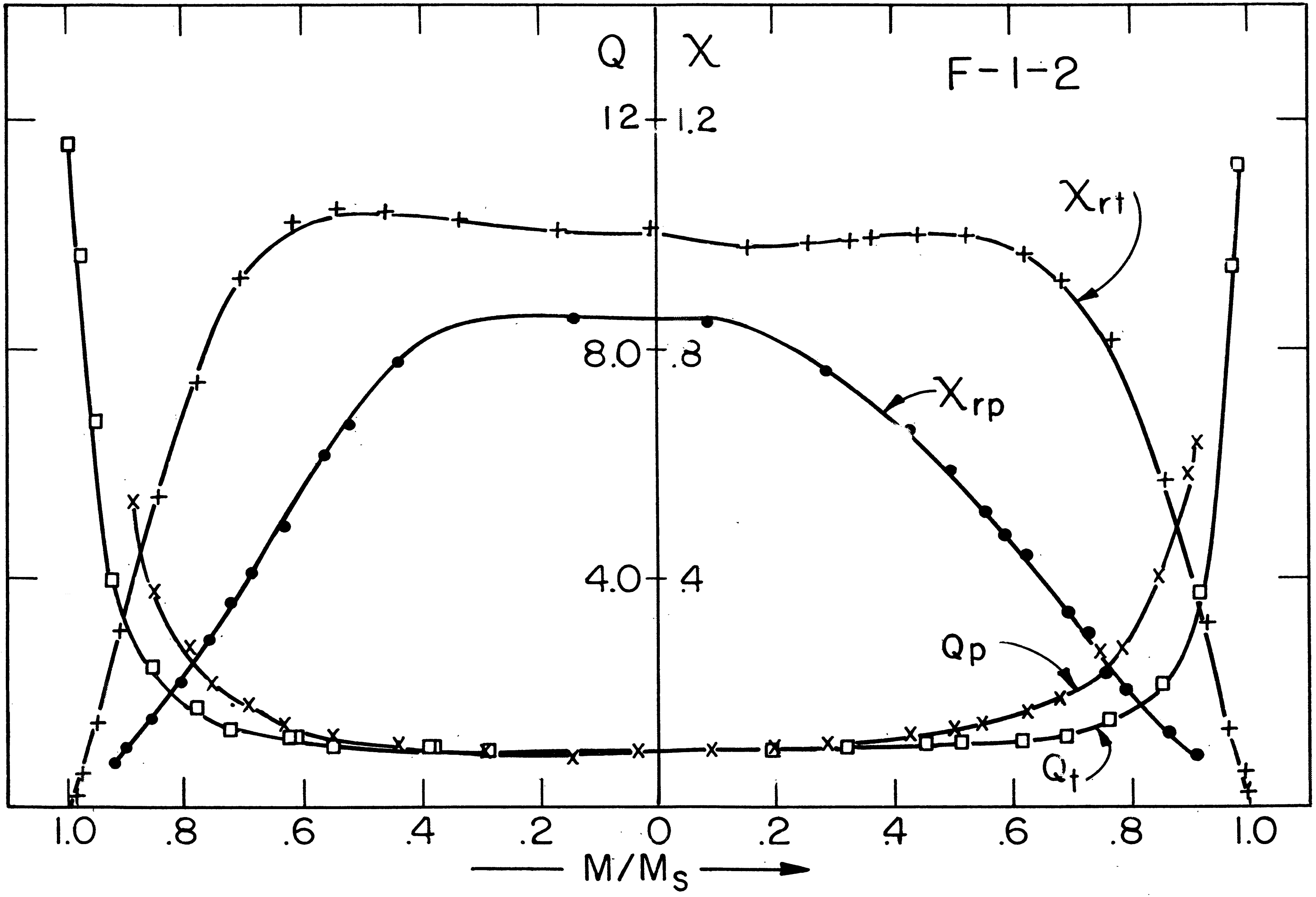

Fig. 2. The Variation of the Normalized Susceptibility and Q with Magnetization for F-1-2.

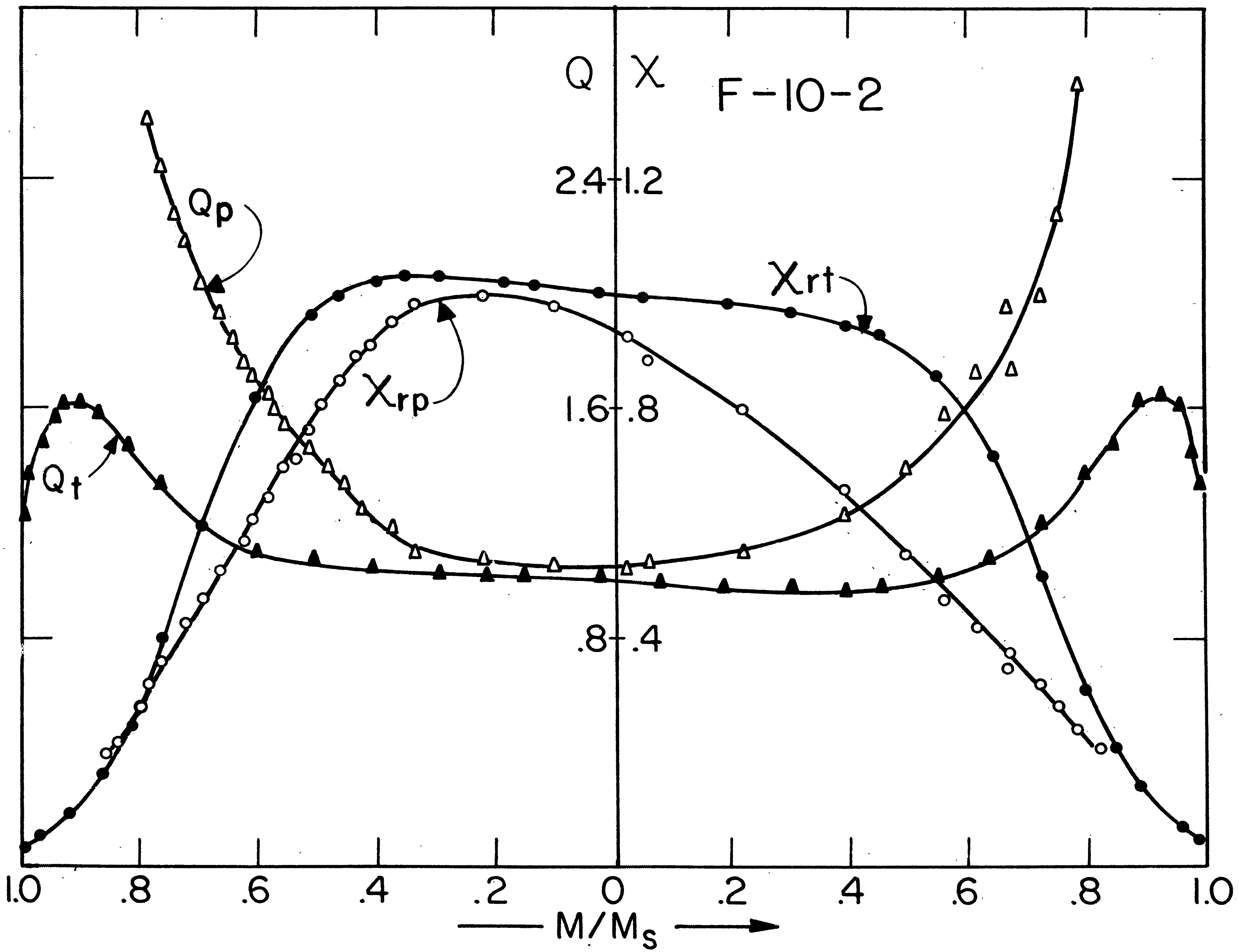

Fig. 3. The Variation of the Normalized Susceptibility and Q with Magnetization for F-10-2.

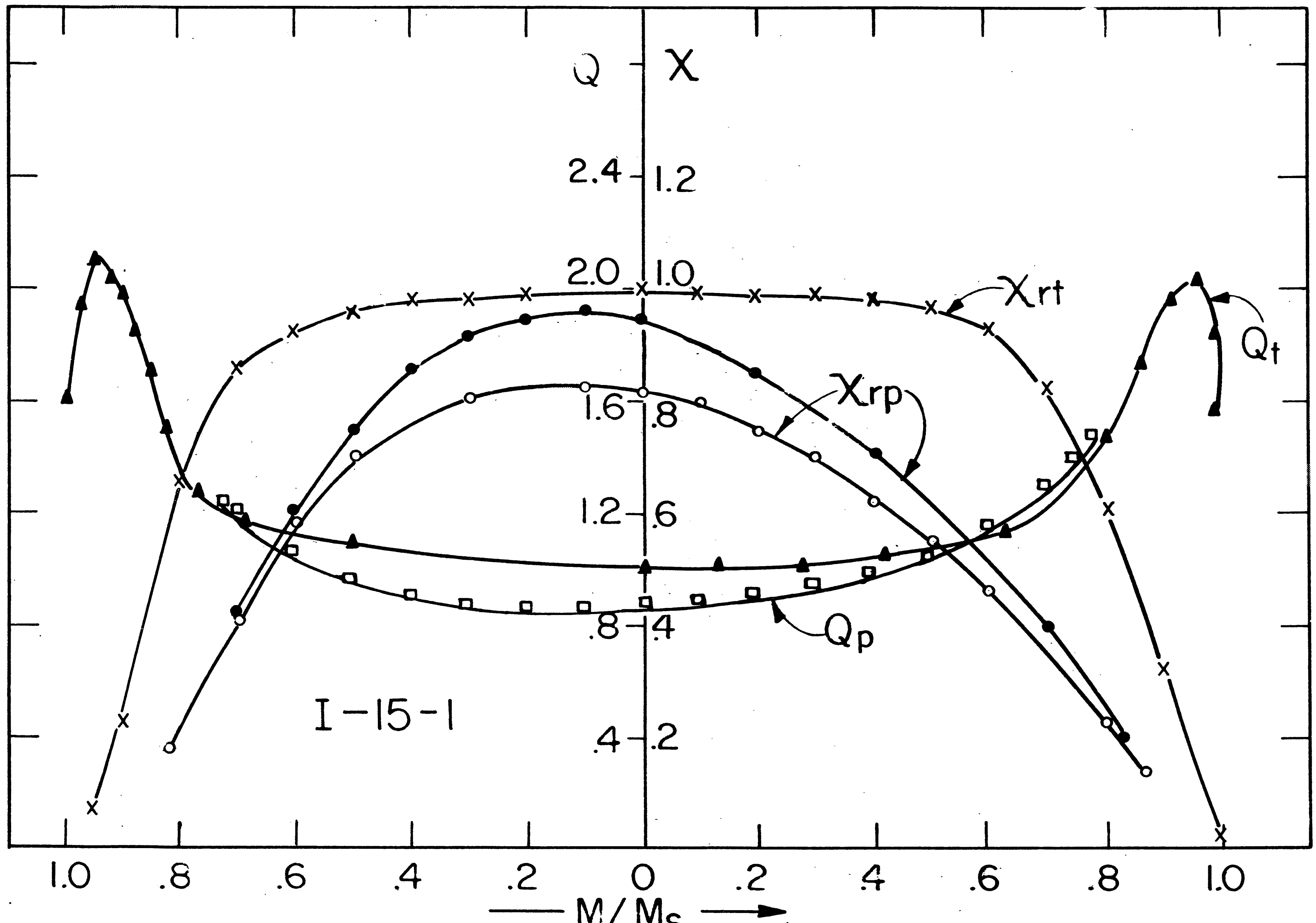

Fig. 4. The Variation of the Normalized Susceptibility and Q with Magnetization for I-15-1.

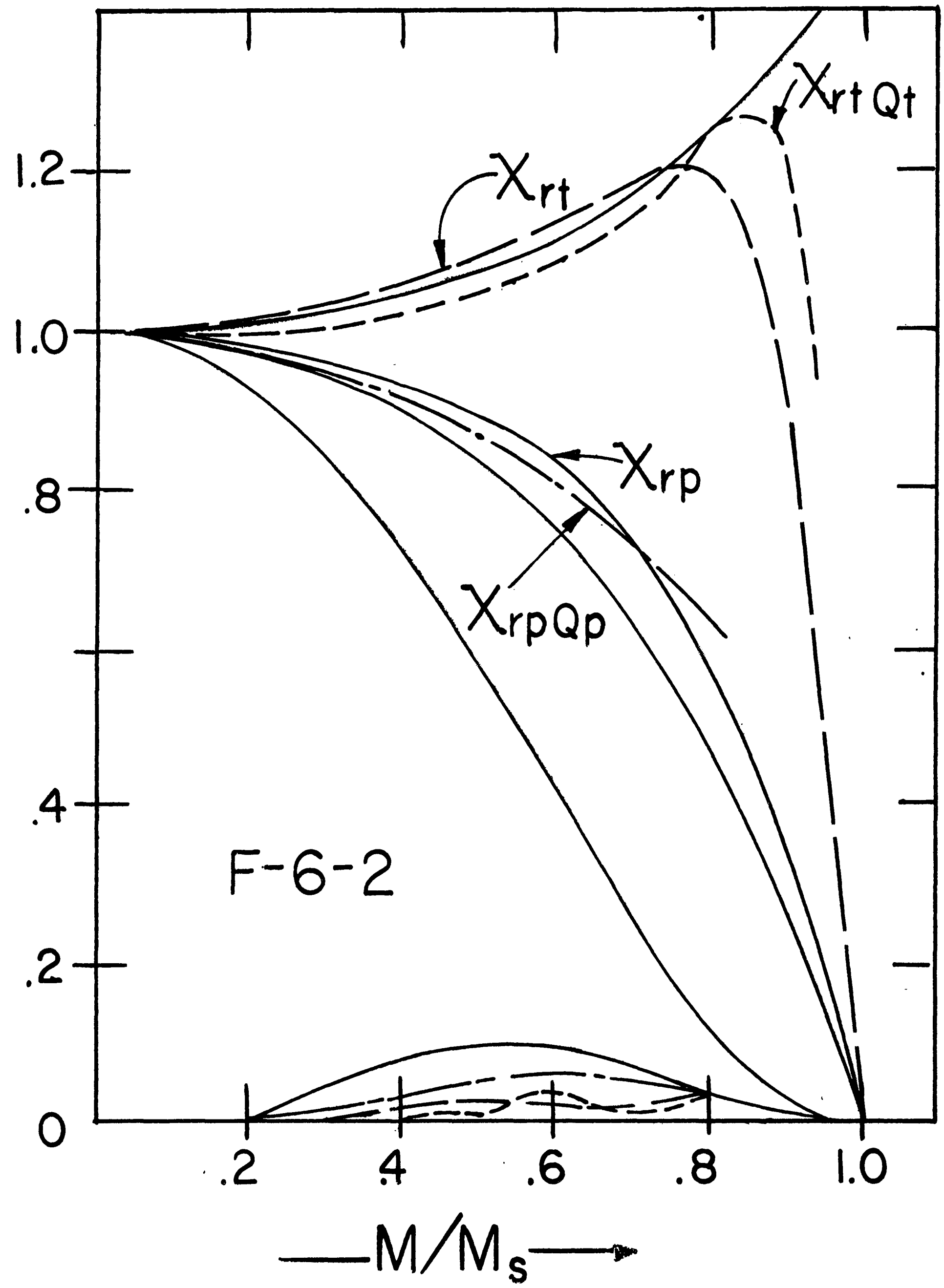

Fig. 5. The Symmetrized χ and χ_Q Curves for F-6-2. The top curves represent the symmetric part and the bottom curves the antisymmetric parts.

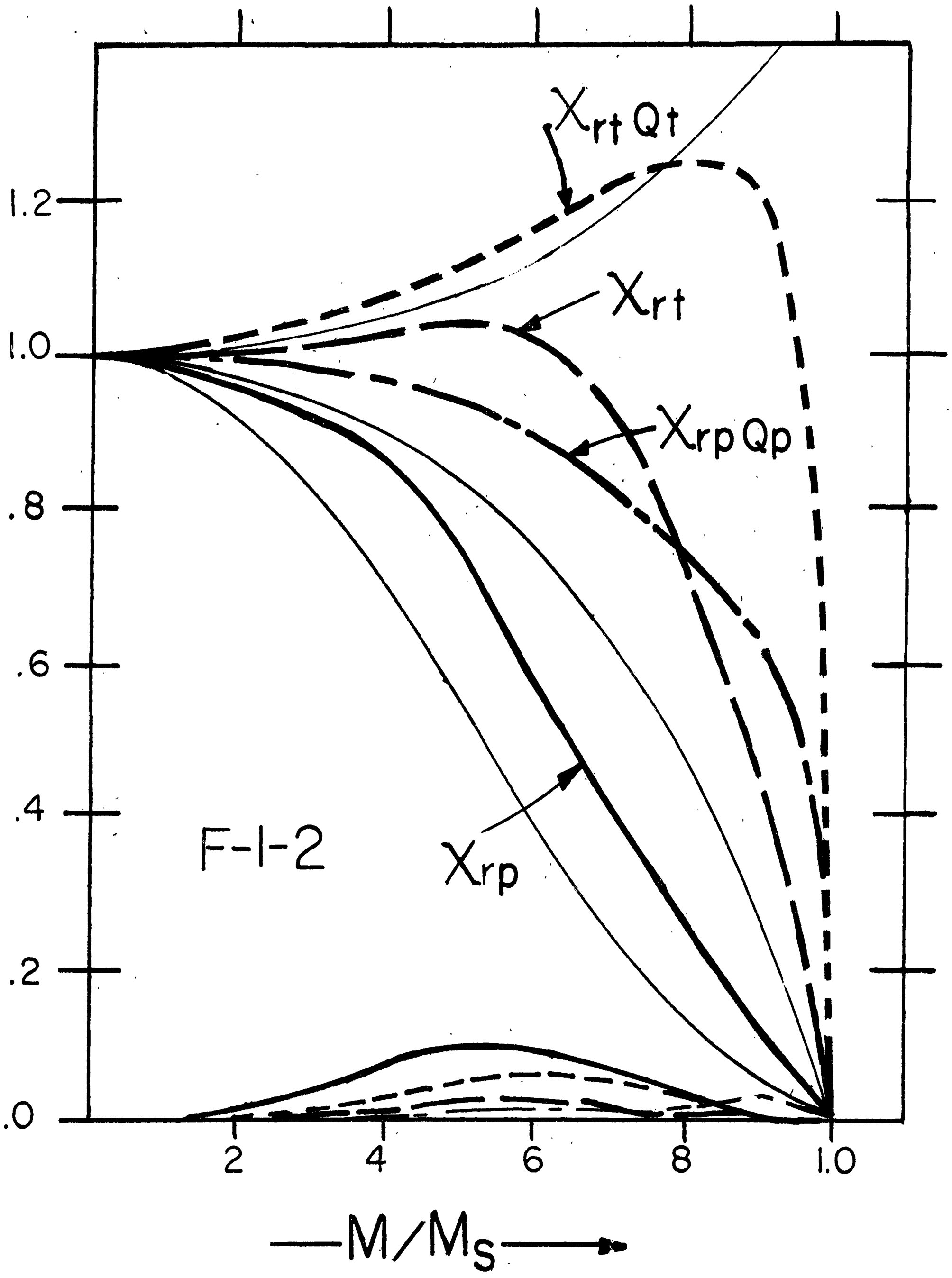

Fig. 6. The Symmetrized χ and χ_Q Curves for F-1-2. The top curves represent the symmetric part and the bottom curves the antisymmetric parts.

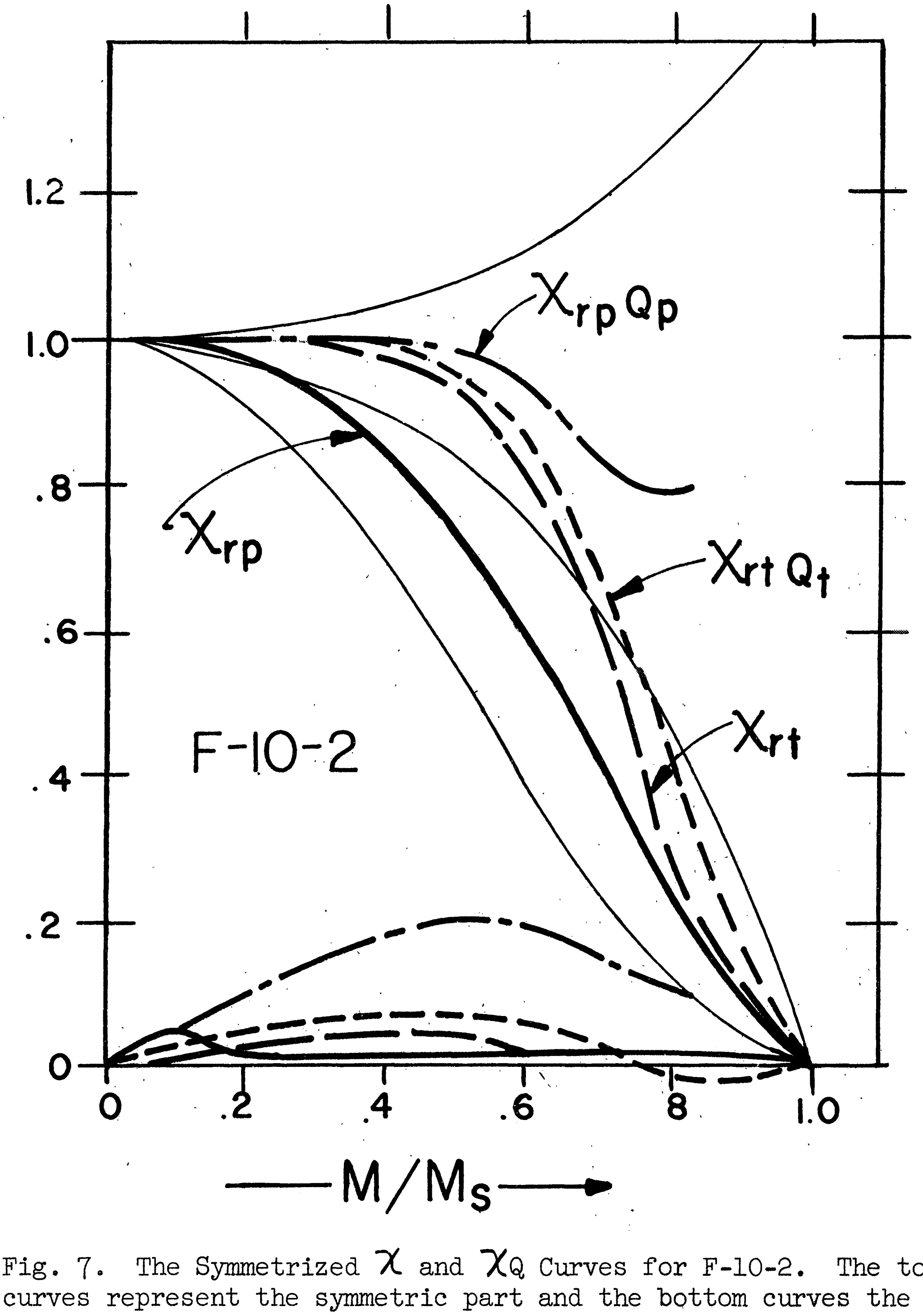

Fig. 7. The Symmetrized χ and χ_Q Curves for F-10-2. The top curves represent the symmetric part and the bottom curves the antisymmetric parts.

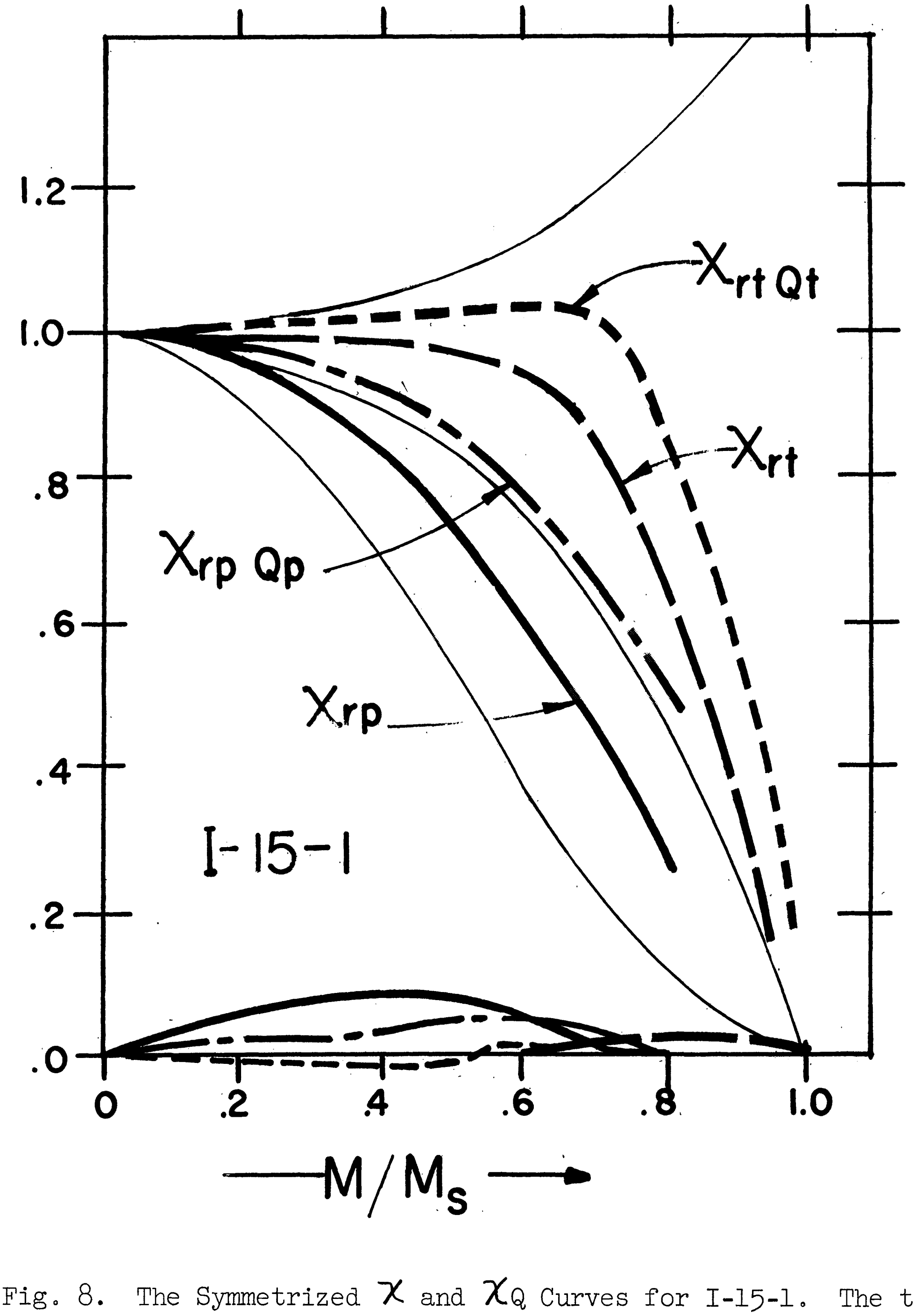

Fig. 8. The Symmetrized χ and χ_Q Curves for I-15-1. The top curves represent the symmetric part and the bottom curves the antisymmetric parts.

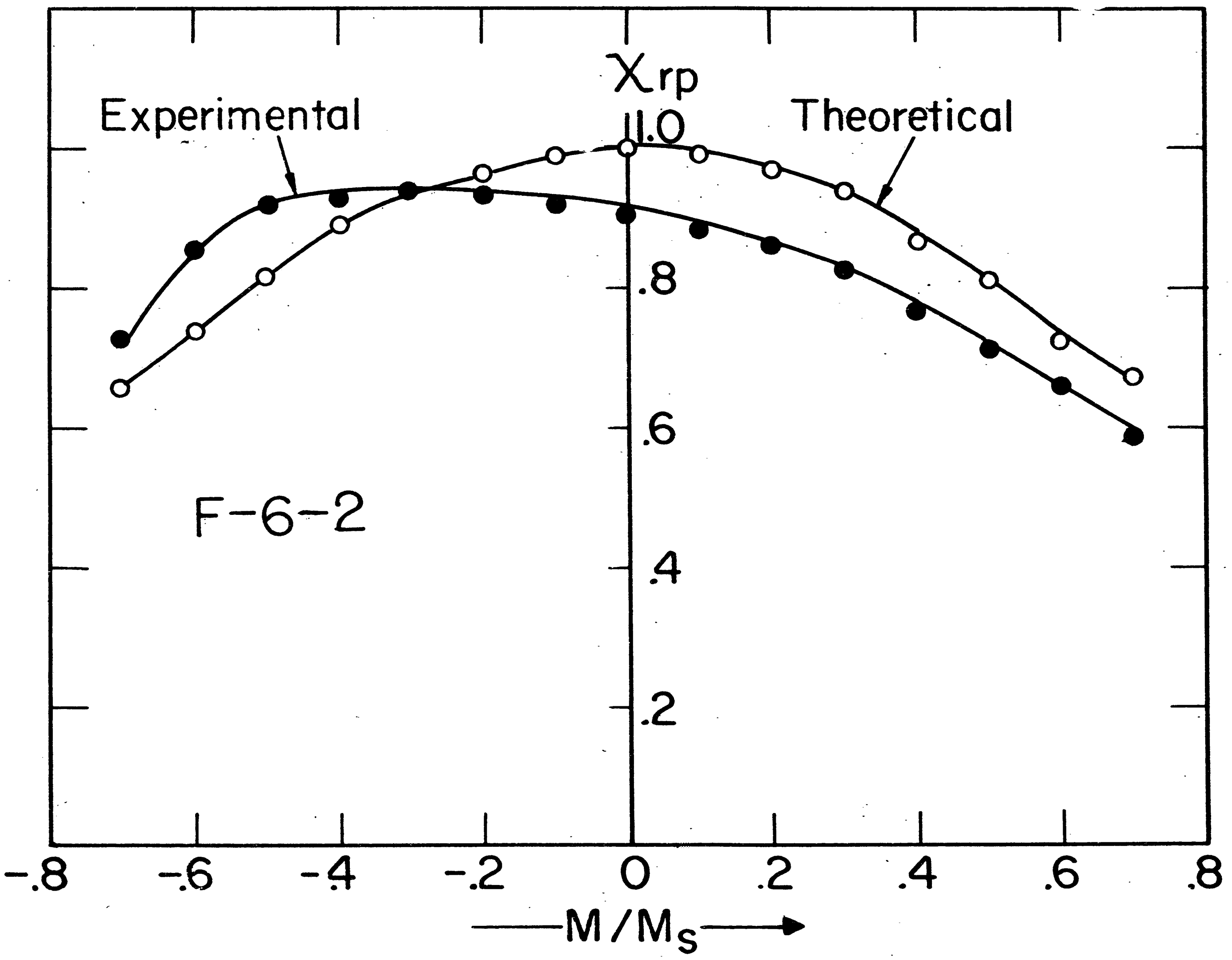

Fig. 9. The solid points represent experimental data, the hollow points parallel susceptibility calculated from the transverse data and Eq. 10 of I.

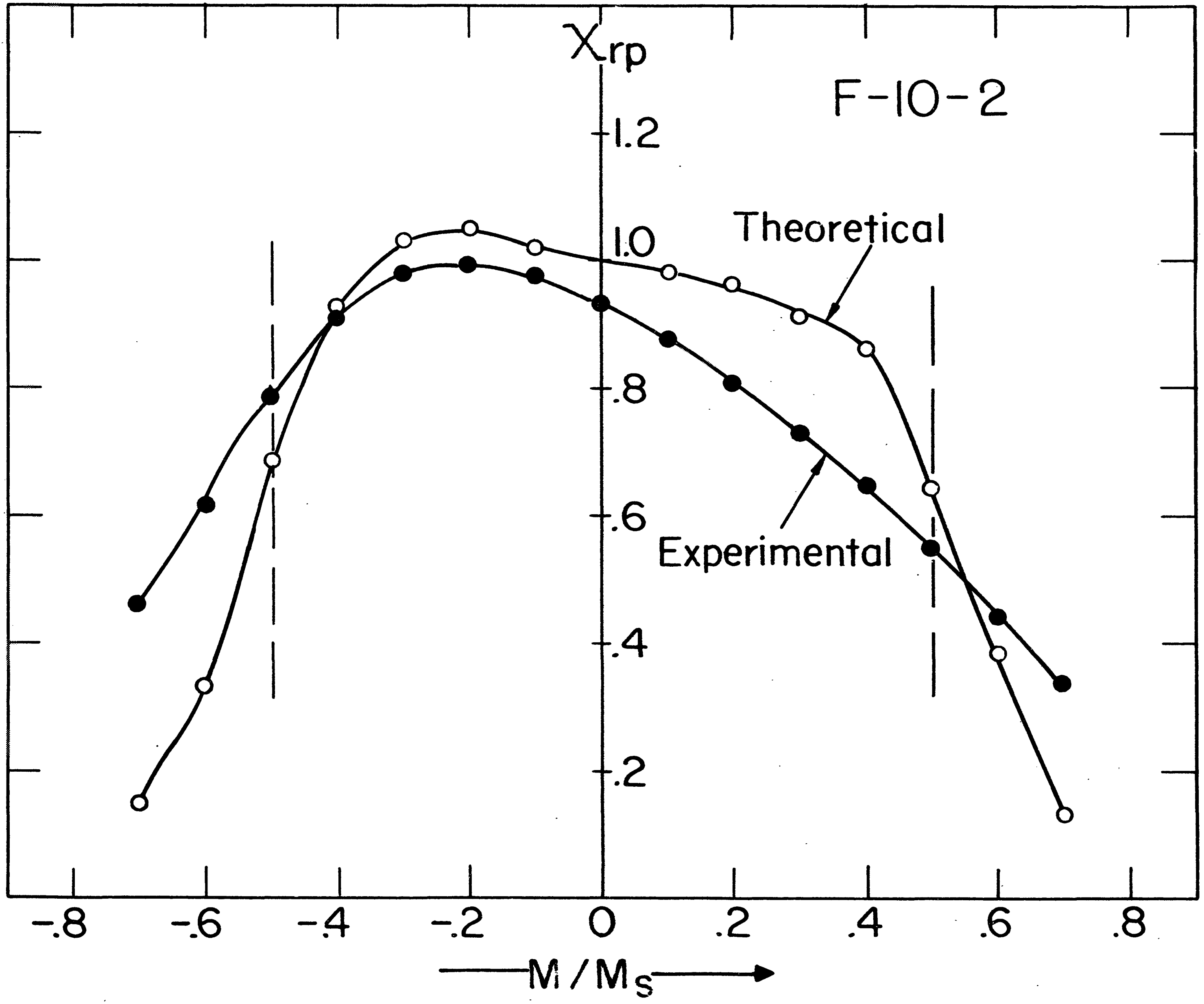

Fig. 10. The solid points represent experimental data, the hollow points the parallel susceptibility calculated from the transverse data and Eq. 13. The dashed curves show the expected approximate limits of validity.