ESTABLISHING THE VALIDITY OF THE MASTER CURVE TECHNIQUE FOR COMPLEX MODULUS DATA REDUCTION

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ABSTRACT

The applicability of the master curve technique for the reduction of temperature- and frequency-dependent complex modulus data to a set of "master" complex modulus curves, which depend on only one variable, is validated for a polyisoprene rubber of shore hardness 55. Using the direct stiffness method, complex Young's modulus data was determined for a sample of the material over 2 narrow frequency bands of 2 octaves each and a wide temperature range of -60C to 100C. Small temperature intervals of 2C at low temperatures rising to 20C at high temperatures were used in the tests. This resulted in two sets of "temperature-dominated" complex modulus data from which smooth, continuous master curves were generated by the application of the master curve technique. The procedure was repeated for a wider test frequency range of 2 decades, the same temperature range but larger temperature increments of 10C at low temperatures rising to 80C at high temperature. This resulted in a "frequency-dominated" complex modulus data set from which master curves were again obtained. It is shown that the master curves obtained from the three data sets correlate quite well.

1. INTRODUCTION

The master curve technique is a well known tool for the reduction of temperature- and frequency-dependent complex modulus data to a set of master curves of modulus and loss factor which depend on a single variable called the reduced frequency or reduced temperature [1-3]. The technique, which is also called the method of reduced variables or the temperature-frequency superposition principle, exploits the inverse relationship of the dependence of complex modulus properties on temperature and frequency to produce a dependence of these properties on a single parameter, the reduced frequency or the reduced temperature, which combines the separate effects of frequency and temperature. Thus complex modulus data obtained over narrow frequency ranges and a wide temperature range, using a single test method, are reduced to sets of single curves of modulus and loss factor which cover several decades of frequency at a specified reference temperature. Similarly, using an appropriate test method or a combination of test methods, complex modulus data can be obtained over a wide frequency range and a small temperature-range or even at constant temperature. From this data, master curves can be again obtained. If, for the material under consideration, the temperature-frequency superposition principle is applicable then the two sets of master curves will be identical.

The use of the master curve technique started on an empirical basis. Subsequently, theoretical models were developed to correlate some of the experimental observations [1]. However, the development and application of the technique has tended to be more empirically orientated. The shift function, which is used for the data reduction process, was for a long time based on the William-Landel-Ferry (WLF) equation. Other forms of shift functions based on the Arrhenius model, statistical method and iterative approach are now in use [3, 4]. It is generally agreed that satisfactory data reduction depends on the use of an appropriate shift function.

Whenever the method of reduced variables is applied, one is confronted by the question of the uniqueness and validity of the generated master curves. Thus, it is often desirable to employ other means to validate the master curves produced. The most direct method of validation is to measure the complex modulus properties at a single temperature, e.g. room temperature, and over a very wide frequency range using a variety of test methods such as stress relaxation, direct stiffness, resonance and ultrasonic methods. However, this is not usually possible as one is often restricted by resources, to the use of one test method. Hence, a different approach is required.

The approach used in the present work for validating the master curve technique is based on an experimental application of a result of the temperature-frequency superposition or equivalence principle. If the principle holds for a given viscoelastic material, then the master curves produced from complex modulus data obtained at constant frequency and varying temperature should be identical to the master curves generated from complex modulus data obtained at constant temperature and varying frequency. The constant frequency data sets are said to be "temperature-dominated" while the constant temperature data sets are said to be "frequency-dominated". Relaxing these two extreme test conditions to become (i) narrow frequency band and many temperature steps, and (ii) wide frequency band and few temperature steps, the sets of complex modulus that will be obtained will still be relatively "temperature-dominated" and "frequency-dominated" respectively. It is shown that for the polyisoprene rubber investigated, the master curves of complex Young's modulus obtained from the temperature-dominated and frequency-dominated data sets are quite similar. The experimental test method employed is the direct stiffness technique.

2. EXPERIMENTAL DETERMINATION OF COMPLEX YOUNG'S MODULUS

2.1 Direct Stiffness Test Method

The direct stiffness method, which is a forced vibration, non-resonance technique for the determination of the complex Young's or shear modulus of polymeric materials [5-7], was used to determine the complex Young's modulus of the polyisoprene rubber investigated. Two samples of the material of 30 mm diameter by 5 mm and 30 mm thick were prepared and bonded to metal discs. Each sample assembly was placed in turn, between the vibration table of an electrodynamic exciter and a rigid termination of theoretically infinite impedance as shown in Figure 1 which also shows the associated measurement and control instrumentation for the experimental tests. The end of the sample connected to the exciter was subjected to controlled sinusoidal displacement excitations of the form $x(t) = Xe^{j\omega t}$. The ratio of the output force $f(t) = F^* e^{j\omega t}$ to the input displacement gave the complex dynamic axial stiffness k^* at the excitation frequency ω. The magnitude k, phase (loss) angle θ, and loss factor η are related to k^* by

$$\begin{aligned} k &= \left|k^*\right| = \left|F^*/X\right| \\ \theta &= \quad k^* = (F^*/X) \\ \eta &= \tan\theta \end{aligned} \tag{1}$$

Thus, the complex dynamic axial stiffness of the sample can be represented as

$$\begin{aligned} k^* &= k^* + jk'' = k'(1 + j\eta) \\ k &= \left|k^*\right| = k'(1 + \eta^2)^{\frac{1}{2}} \end{aligned} \tag{2}$$

2.2 Derivation of Complex Young's Modulus

Due to the restraints imposed on the bonded ends of a sample, subjected to tension-compression deformation, multiplying the complex stiffness k^* by the factor L/A, where L is length and A is cross-sectional area of a prismatic sample gives an apparent complex Young's modulus, E_a^*. The true and apparent magnitudes E and E_a of the true and apparent complex Young's moduli are related by [2,8]

$$E_a = E(1 + \beta S^2) \ ; \ E_a = kL/A \tag{3}$$

where β is a numerical constant which has values of $1.5 \leq \beta \leq 2.0$ that depend on the filler content of the elastomer, and S is a shape factor defined as,

$$S = D/4L \tag{4}$$

for a solid cylindrical element of diameter D and length L.

Thus, the true complex Young's modulus E^* is related to the measured complex extensional stiffness k^* by

$$E^* = E_a^*/(1 + \beta S^2) = k^*L/(1 + \beta S^2)A \tag{5}$$

where

$$E = |E^*| = kL/(1 + BS^2)A \tag{6}$$

$$\eta_E = \eta$$

3. COMPLEX YOUNG'S MODULUS DATA AND MASTER CURVES.

Using the procedures described above, the complex Young's modulus of the polyisoprene rubber was determined under three test frequency conditions, namely;

(1) Narrow band, low frequency tests (10 to 40 Hz)
(2) Narrow band, intermediate frequency tests (100 to 400 Hz)
(3) Wide frequency band tests (10 to 1000 Hz)

The complex Young's modulus data obtained under these test conditions and the resultant master curves are described in the following. In reducing the data to master curves, the modulus data was firstly shifted vertically using the relation

$$E_r = (T_r/T)E \tag{7}$$

where E is the Young's modulus at experimental temperature T, E_r is the reduced Young's modulus at the reference temperature T_r, and density variations are assumed to be negligible. The shift factors used were of the general form

$$\log\alpha_T = \varphi(T - T_r) \tag{8}$$

where the forms of the function φ were determined directly from the measured data.

3.1 Narrow Band, Low Frequency Data

For the narrow band, low frequency tests, the experimental frequency was from 10 to 40 Hz, that is 2 octaves, while the temperature range was from −60C to 100C. The temperature steps were 2C between −60C and −30C, about 3C between −30C and −10C, 10C between −10C and 20C, and 20C for test temperatures between 20C and 100C. This resulted in 30 test temperatures. The number of frequency points was 6 per data set. Thus, the total number of Young's modulus and loss factor pairs of data obtained was 180 as shown in Table 1. These data sets are certainly "temperature-dominated". Figure 2 shows the wicket plot of log (loss factor) versus log (modulus) for the data. The low temperature (below −50C) data seems to be subject to relatively higher random errors whereas the high temperature data (above 20C) seems to be affected by some systematic effects. Using the master curve technique, in its general empirical form, master curves of Young's modulus and loss factor were produced from this data at a reference temperature of −40C. The shift function used for the data reduction process is shown in Figure 3. It was estimated numerically from the data using a computerized data shifting process. The resultant master curves are shown in Figure 4. It can be seen that the data scatter is low but it is significant for the loss factor curve at low temperatures.

3.2 Narrow Band, Intermediate Frequency Data

The test conditions for this case are quite similar to those of the previous case. The only difference is that the frequencies are ten times higher than those of the narrow band, low frequency data. Thus, the frequency range for the narrow band, intermediate frequency tests is 100 to 400 Hz (2 octaves) with unequal frequency increments resulting in a total of 6 frequency points per data set. The test temperatures and temperature steps are excactly identical to those of the previous case. Thus, 180 pairs of temperature-dominated data were again obtained as shown in Table 2. The wicket plot of the data is shown in Figure 5. It can be seen that the data scatter is generally small being higher for temperatures greater than 40C. By repeated data shifting process a "best" estimate of the shift factor curve was obtained at a reference temperature of -40C as shown in Figure 6. Using this curve the complex Young's modulus data was reduced to master curves as shown in Figure 7. It can be seen that the data scatter is quite small being relatively more pronounced for the loss factor data at higher temperatures.

3.3 Wide Frequency Band Data

The frequency range of the wide frequency band tests was wider than the previous cases being from 10 to 1000 Hz (2 decades). The frequency steps were about 12 per decade giving 23 frequency steps in total. The temperature range used for the tests was again from -60C to 100C but the temperature steps were higher being -10C at very low temperatures and rising to 80C at high temperature. The number of test temperatures used was 6. Thus, the total number of pairs of data was 138 as shown in Table 3. These data sets are relatively more "frequency-dominated" than in the previous cases. Figure 8 shows the wicket plot for these data sets. It can be seen that the data scatter due to random errors is small. However the 100C data set seems to be subjected to some systematic errors as it is somewhat removed from the general body of data. By means of the master curve technique, the data was reduced to master curves of Young's modulus and loss factor at a reference temperature of -40C. The shift factor curve used for the data reduction is shown in Figure 9. The master curves obtained are shown in Figure 10. It can be seen that random data scatter is small but there may be some systematic errors with the high temperature (low reduced frequency) data.

3.4 Comparison of Master Curves

The three sets of master curves of complex Young's modulus obtained are compared with one another as shown in Figure 11. Except for some slight discrepancies which occur in the loss factor master curves as high reduced frequencies, it can be seen that the master curves correlate reasonably well within the limits of data measurement and processing errors. This implies that whether temperature-dominated or frequency-dominated data is used, the master curves generated will be very similar and unique. Thus, it can be concluded that the temperature-frequency superposition is valid for the reduction of temperature-and frequency-dependent complex modulus data of polyisoprene rubber to master curves.

4. CONCLUSIONS

The master curve methodology is valid for the reduction of complex modulus data to master curves provided the material under consideration is thermorheologically simple. It has been demonstrated that the application of the master curve technique for the reduction of complex Young's modulus data of the polyisoprene rubber investigated is valid. Thus, it can be inferred that this material is thermorheologically simple. When it is uncertain whether a material is thermorheologically simple, such a validity test, as demonstrated in this paper, might prove useful.

5. ACKNOWLEDGEMENTS

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TABLE 1 : COMPLEX YOUNG'S MODULUS DATA FOR POLYISOPRENE RUBBER
(NARROW BAND, LOW FREQUENCY DATA)

	f= 10Hz		f= 15Hz		f= 20Hz		f= 25Hz		f= 30Hz		f= 40Hz	
T(C)	E(MPa)		E(MPa)		E(MPa)		E(MPa)		E(MPa)		E(MPa)	
-60	2042.1	.30	2087.4	.27	2132.8	.25	2178.2	.24	2223.6	.21	2268.9	.21
-58	1769.5	.37	1821.6	.34	1873.6	.30	1925.7	.31	1977.7	.30	2029.8	.28
-56	1165.0	.53	1234.0	.45	1303.1	.50	1396.5	.39	1538.4	.43	1665.3	.36
-54	806.9	.75	851.8	.64	896.6	.63	956.4	.52	1050.6	.58	1333.0	.55
-52	485.4	1.16	516.7	.95	548.0	.93	620.0	.83	697.0	.82	828.7	.88
-50	270.1	1.52	331.8	1.42	374.5	1.31	415.9	1.28	457.9	1.24	523.7	1.17
-48	137.3	1.33	176.4	1.43	211.3	1.43	237.4	1.36	261.7	1.39	326.7	1.43
-46	100.6	1.39	113.5	1.38	146.9	1.31	169.0	1.39	198.3	1.48	230.5	1.53
-44	55.2	1.13	70.4	1.24	83.6	1.31	97.1	1.30	112.4	1.36	137.8	1.39
-42	38.5	1.06	48.6	1.17	58.7	1.25	67.4	1.31	75.1	1.31	85.6	1.40
-30	26.3	.99	33.5	1.08	39.4	1.17	45.4	1.22	50.6	1.27	61.2	1.30
-38	19.4	.87	23.7	.97	27.6	1.02	30.7	1.06	34.7	1.13	41.8	1.08
-36	14.9	.78	17.9	.86	20.6	.91	23.0	.95	25.5	.98	29.9	1.02
-34	11.5	.65	13.6	.73	15.5	.80	17.2	.84	19.0	.87	22.7	.91
-32	10.3	.60	12.1	.68	13.8	.73	15.4	.77	16.7	.81	19.6	.87
-30	8.9	.52	10.2	.59	11.5	.65	12.6	.69	13.6	.72	15.5	.78
-28	7.6	.41	8.5	.48	9.3	.52	10.0	.56	10.7	.58	12.0	.63
-25	6.9	.33	7.7	.40	8.3	.44	8.8	.48	9.4	.51	10.4	.57
-23	6.3	.28	6.8	.33	7.2	.37	7.6	.41	8.1	.42	8.8	.46
-20	6.2	.22	6.3	.25	6.8	.29	7.0	.32	7.3	.35	8.2	.40
-17	5.6	.17	5.8	.21	6.1	.24	6.3	.26	6.5	.28	6.9	.32
-13	5.4	.13	5.5	.16	5.8	.18	5.9	.20	6.0	.22	6.4	.24
-10	5.4	.10	5.6	.13	5.7	.13	5.8	.15	5.9	.17	6.1	.18
0	5.2	.07	5.2	.08	5.3	.08	5.4	.09	5.5	.10	5.6	.11
10	4.9	.06	5.0	.06	5.1	.06	5.1	.07	5.2	.08	5.2	.08
20	4.9	.05	4.9	.05	4.9	.05	5.0	.06	5.0	.06	5.2	.06
40	4.3	.06	4.4	.06	4.5	.06	4.6	.06	4.6	.06	4.7	.06
60	4.1	.06	4.2	.06	4.2	.06	4.3	.06	4.4	.06	4.5	.06
80	3.9	.06	4.0	.06	4.1	.06	4.2	.06	4.3	.06	4.3	.05
100	3.8	.06	3.9	.06	4.0	.06	4.1	.06	4.1	.05	4.2	.06

TABLE 2 : COMPLEX YOUNG'S MODULUS DATA FOR POLYISOPRENE RUBBER
(NARROW BAND, INTERMEDIATE FREQUENCY DATA)

	f=100Hz		f=150Hz		f=200Hz		f=250Hz		f=300Hz		f=400Hz	
T(C)	E(MPa)		E(MPa)		E(MPa)		E(MPa)		E(MPa)		E(MPa)	
-60	2495.8	.13	2413.3	.13	2565.5	.12	2526.0	.11	2525.1	.10	2678.4	.10
-58	2290.0	.18	2188.1	.15	2353.3	.16	2361.3	.14	2361.0	.14	2505.5	.14
-56	1866.4	.30	1960.5	.27	2111.2	.26	2133.3	.22	2164.0	.22	2291.9	.20
-54	1544.9	.38	1781.1	.35	1923.2	.32	1974.6	.28	2001.1	.27	2143.2	.25
-52	1164.8	.60	1375.4	.56	1530.4	.50	1609.4	.44	1660.2	.41	1814.8	.38
-50	848.1	.85	967.0	.80	1117.6	.71	1207.7	.66	1279.2	.62	1430.4	.56
-48	513.8	1.40	669.1	1.05	783.1	.97	875.9	.88	955.0	.83	1094.2	.75
-46	403.9	1.45	531.9	1.18	637.5	1.08	713.7	1.01	777.5	.95	896.4	.88
-44	236.1	1.56	313.1	1.42	380.8	1.35	431.5	1.29	481.6	1.23	571.7	1.17
-42	159.4	1.56	212.3	1.51	258.0	1.48	297.7	1.43	332.3	1.39	401.7	1.35
-30	99.4	1.45	131.2	1.50	158.8	1.52	183.4	1.53	207.1	1.51	253.3	1.49
-38	68.6	1.41	88.7	1.45	106.5	1.46	123.7	1.49	138.9	1.49	168.3	1.53
-36	48.9	1.27	62.6	1.33	75.2	1.40	85.7	1.42	96.5	1.44	116.2	1.48
-34	35.5	1.13	45.0	1.22	53.4	1.27	60.8	1.31	68.3	1.34	82.3	1.39
-32	29.3	1.07	36.9	1.15	43.3	1.21	49.1	1.25	54.6	1.28	65.0	1.32
-30	23.2	.96	28.6	1.04	33.1	1.10	37.5	1.14	41.7	1.16	49.6	1.22
-28	16.7	.81	20.3	.88	23.2	.92	26.1	.96	28.8	.99	34.0	1.05
-25	14.1	.73	16.7	.81	19.1	.86	21.5	.90	23.3	.93	27.4	.97
-23	11.4	.61	13.3	.68	14.9	.72	16.5	.76	17.8	.78	20.7	.84
-20	9.9	.55	11.5	.64	12.8	.69	14.2	.72	15.2	.75	17.7	.78
-17	8.4	.45	9.4	.52	10.5	.56	11.3	.60	12.2	.61	14.0	.68
-13	7.4	.36	8.1	.42	8.9	.45	9.6	.50	10.3	.52	11.9	.59
-10	6.8	.31	7.4	.38	8.0	.41	8.4	.45	8.8	.48	9.9	.49
0	5.9	.16	6.1	.19	6.5	.22	6.6	.23	7.0	.24	7.7	.28
10	5.6	.09	5.7	.12	5.9	.14	6.1	.14	6.3	.14	6.8	.15
20	5.4	.07	5.5	.07	5.6	.08	5.9	.08	5.8	.09	6.1	.11
40	4.9	.06	5.1	.06	5.1	.06	5.3	.06	5.5	.06	5.8	.06
60	4.7	.06	4.8	.06	5.0	.05	5.1	.06	5.3	.05	5.3	.06
80	4.5	.06	4.5	.07	4.7	.06	4.9	.05	5.1	.06	5.4	.06
100	4.4	.06	4.5	.06	4.6	.05	4.7	.06	4.9	.06	5.0	.06

TABLE 3 : COMPLEX YOUNG'S MODULUS DATA FOR POLYISOPRENE RUBBER
(WIDE FREQUENCY BAND DATA)

	T=-60C		T=-50C		T=-40C		T=-20C		T= 20C		T=100C	
f(Hz)	E(MPa)		E(MPa)		E(MPa)		E(MPa)		E(MPa)		E(MPa)	
10	2042.1	.30	270.1	1.52	26.3	.99	6.2	.22	4.9	.05	3.8	.06
15	2087.4	.27	331.8	1.42	33.5	1.08	6.3	.25	4.9	.05	3.9	.06
20	2132.8	.25	374.5	1.31	39.4	1.17	6.8	.29	4.9	.05	4.0	.06
25	2178.2	.24	415.9	1.28	45.4	1.22	7.0	.32	5.0	.06	4.1	.06
30	2223.6	.21	457.9	1.24	50.6	1.27	7.3	.35	5.0	.06	4.1	.05
40	2268.9	.21	523.7	1.17	61.2	1.30	8.2	.37	5.2	.06	4.2	.06
50	2314.3	.19	589.5	1.11	72.1	1.34	8.5	.41	5.2	.06	4.2	.06
60	2359.7	.16	659.5	1.05	78.8	1.36	8.9	.44	5.3	.07	4.3	.05
70	2405.1	.15	729.5	1.00	85.6	1.36	8.9	.45	5.3	.07	4.3	.06
80	2450.5	.15	749.2	.95	89.7	1.34	9.3	.46	5.3	.07	4.3	.06
90	2495.8	.15	769.0	.90	91.8	1.40	9.7	.49	5.3	.07	4.4	.06
100	2495.8	.13	848.1	.85	99.4	1.45	9.9	.54	5.4	.07	4.4	.06
150	2413.3	.13	967.0	.80	131.2	1.50	11.5	.59	5.5	.07	4.5	.06
200	2565.5	.12	1117.6	.71	158.8	1.52	12.8	.62	5.6	.08	4.6	.05
250	2526.0	.11	1207.7	.66	183.4	1.53	13.9	.67	5.6	.08	4.6	.06
300	2525.1	.10	1279.2	.62	207.1	1.51	14.7	.71	5.6	.09	4.9	.06
400	2678.4	.10	1430.4	.56	253.3	1.49	16.6	.76	5.7	.11	5.0	.06
500	2669.0	.10	1524.6	.52	294.2	1.44	18.2	.78	6.2	.12	5.2	.06
600	2779.1	.10	1610.1	.48	333.6	1.43	19.6	.82	6.3	.11	5.1	.06
700	2741.9	.10	1592.9	.47	375.6	1.37	21.1	.83	6.3	.12	5.7	.04
800	3055.3	.10	1740.0	.45	409.7	1.38	22.1	.87	6.1	.13	5.4	.06
900	2844.9	.08	1712.3	.43	448.6	1.30	23.1	.89	6.3	.15	5.7	.05
1000	3024.5	.07	1858.5	.41	493.1	1.28	24.2	.93	6.6	.16	5.9	.06

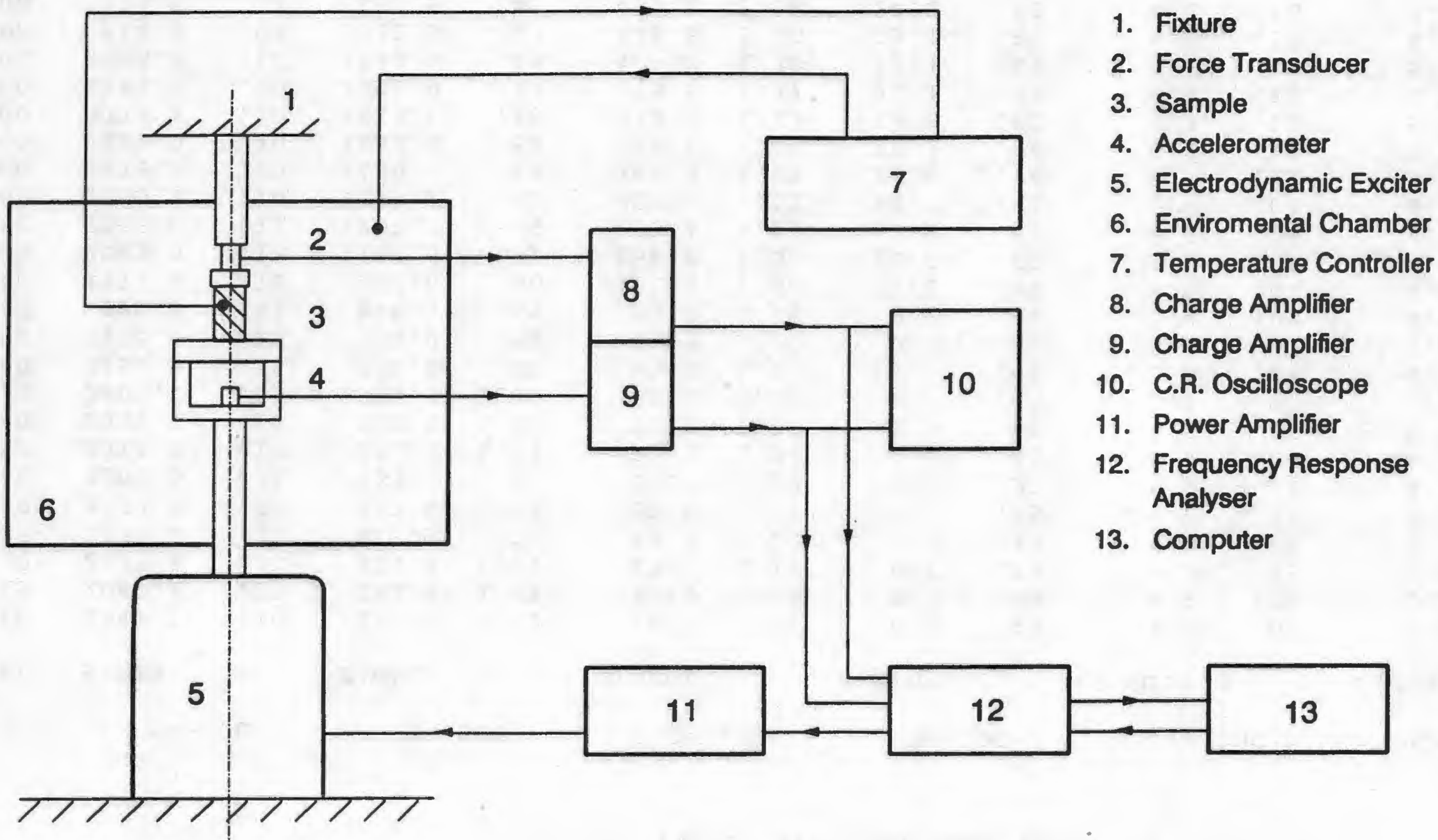

FIGURE 1 : SCHEMATIC DIAGRAM OF DIRECT COMPLEX STIFFNESS TEST CONFIGURATION

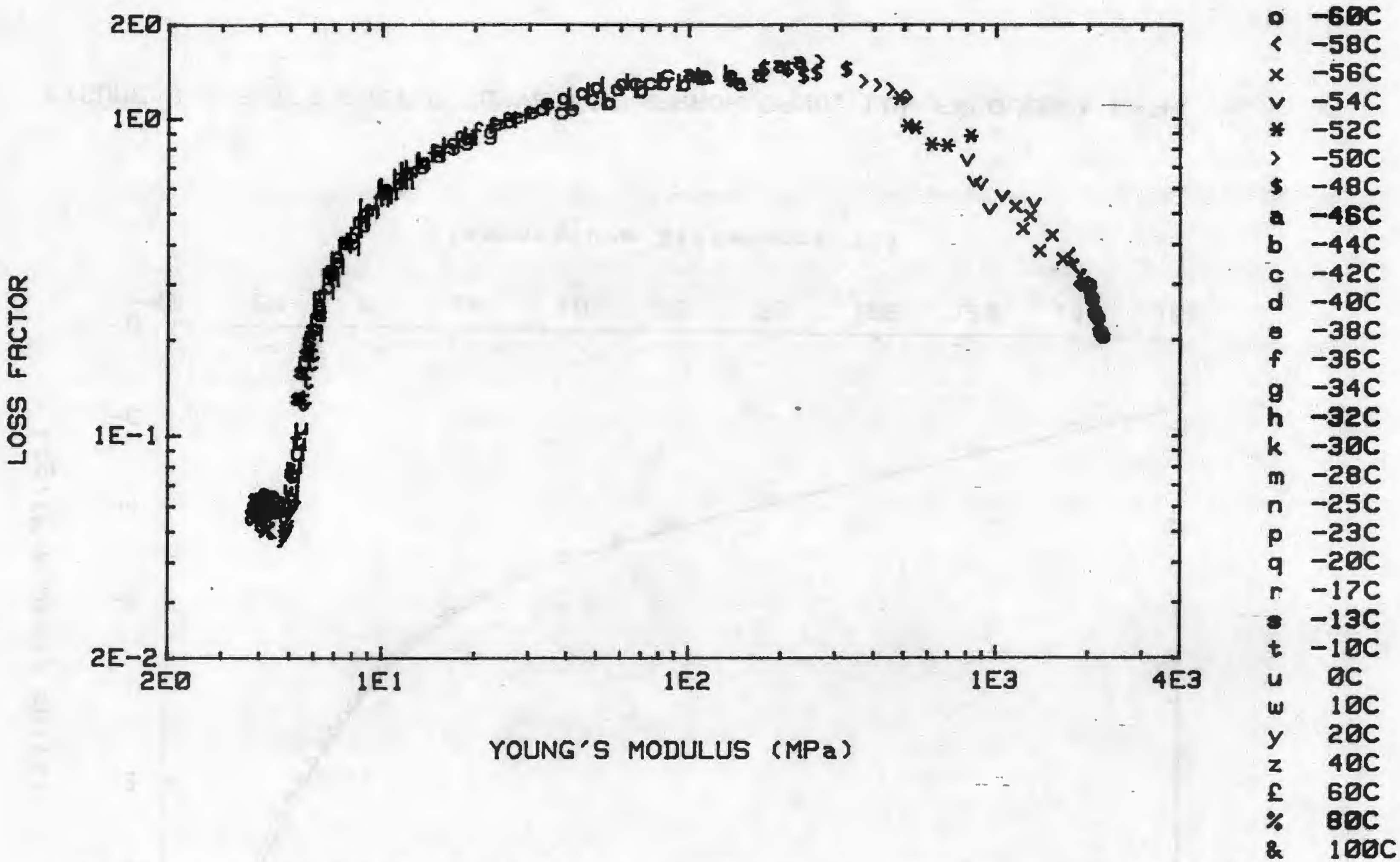

FIGURE 2 : LOSS FACTOR VS. MODULUS (WICKET PLOT) FOR POLYISOPRENE RUBBER (NARROW BAND, LOW FREQUENCY DATA)

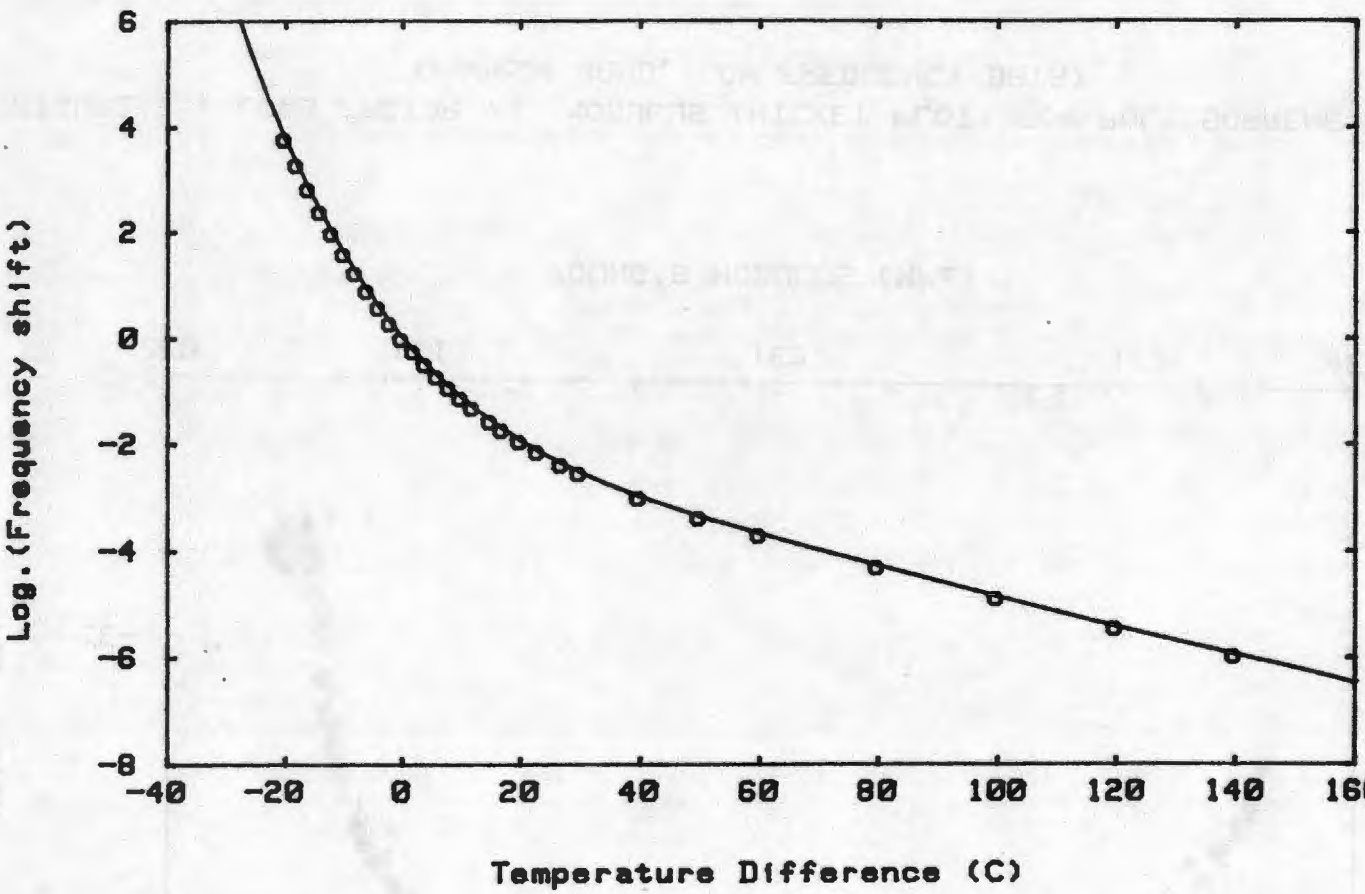

FIGURE 3 : SHIFT FACTOR CURVE FOR NARROW BAND, LOW FREQUENCY DATA

DBC-13

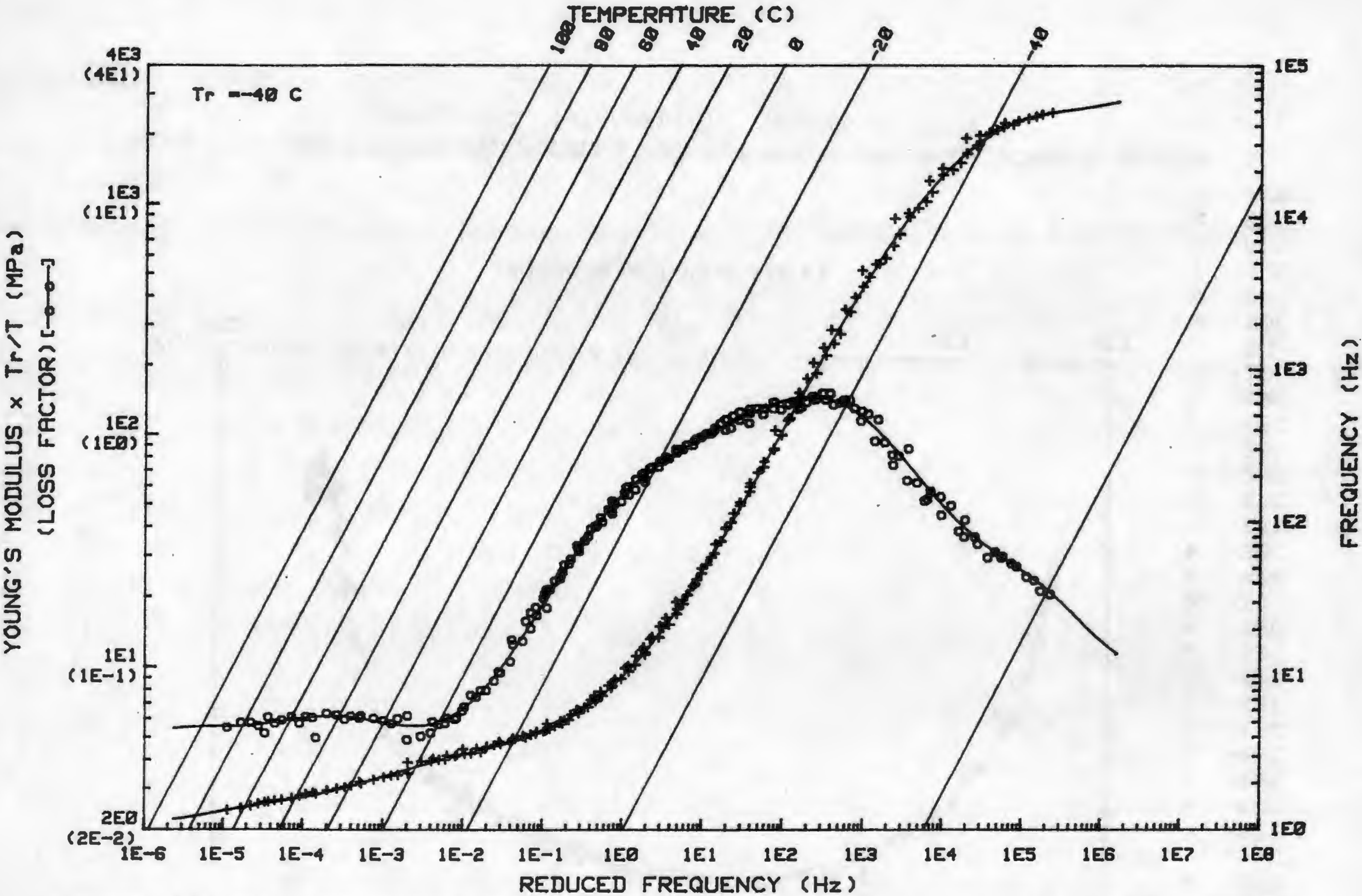

FIGURE 4 : INTERNATIONAL PLOT OF COMPLEX YOUNG'S MODULUS FOR POLYISOPRENE RUBBER
(NARROW BAND, LOW FREQUENCY DATA)

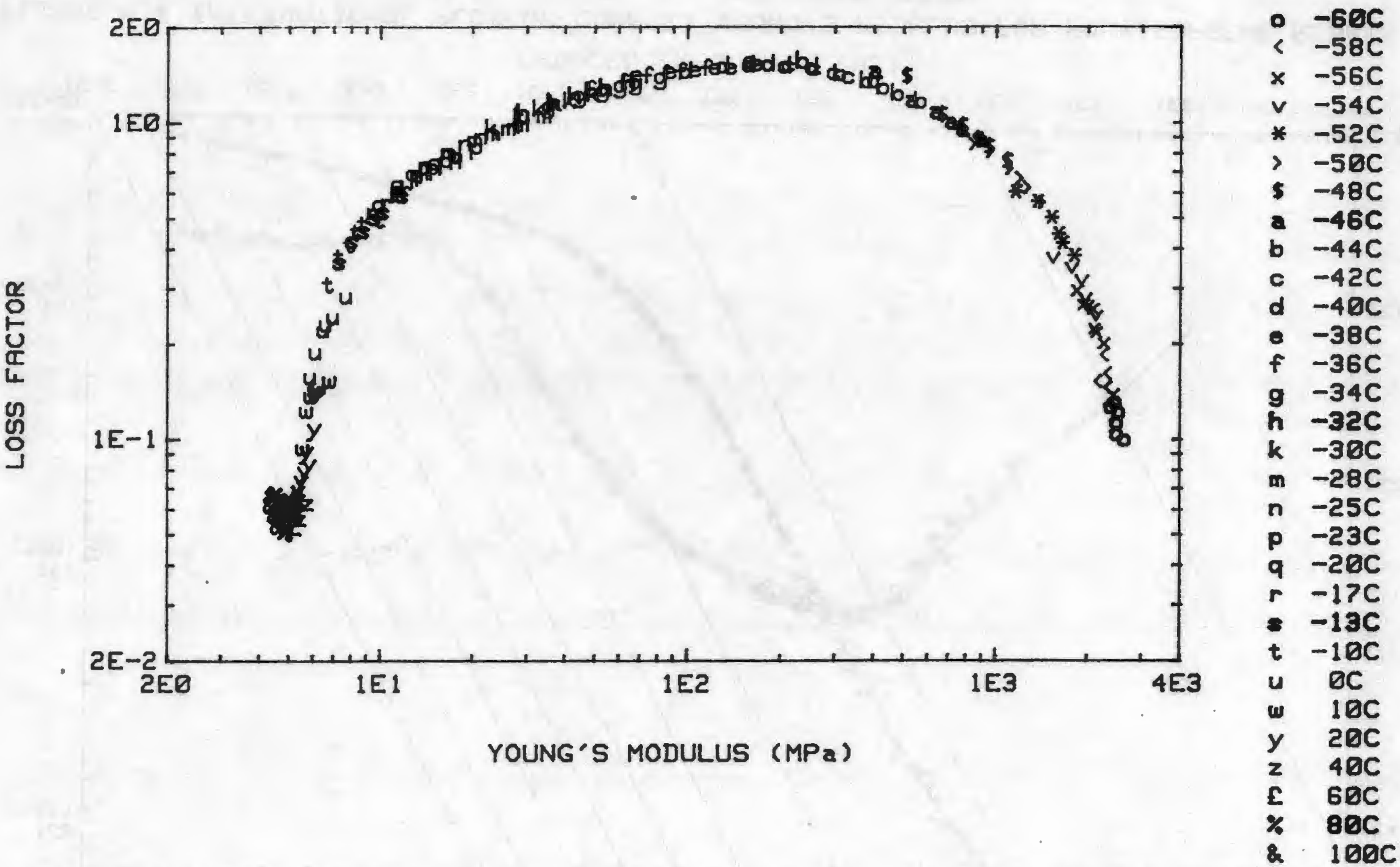

FIGURE 5 : LOSS FACTOR VS. MODULUS (WICKET PLOT) FOR POLYISOPRENE RUBBER
(NARROW BAND, INTERMEDIATE FREQUENCY DATA)

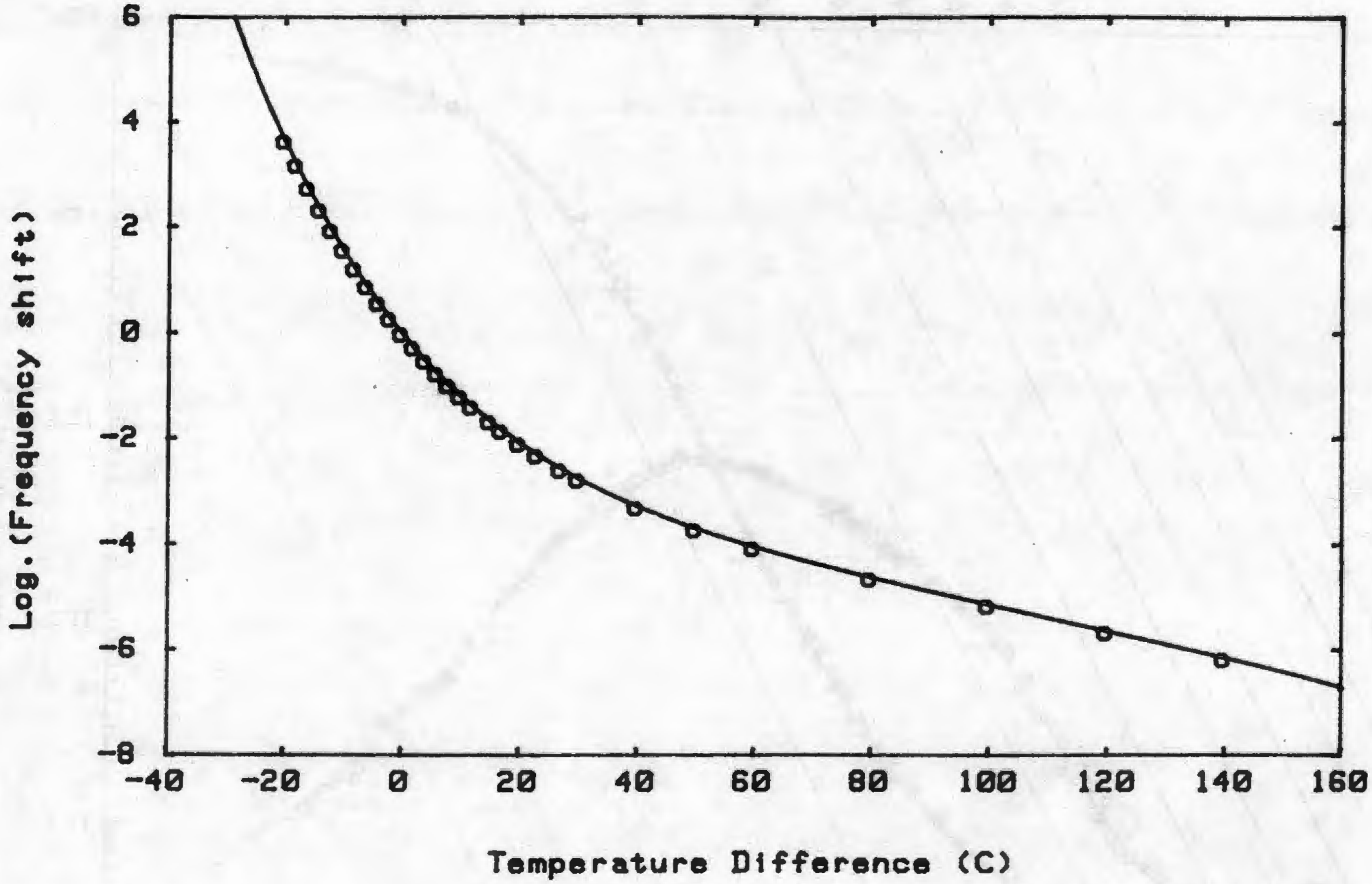

FIGURE 6 : SHIFT FACTOR CURVE FOR NARROW BAND, INTERMEDIATE FREQUENCY DATA

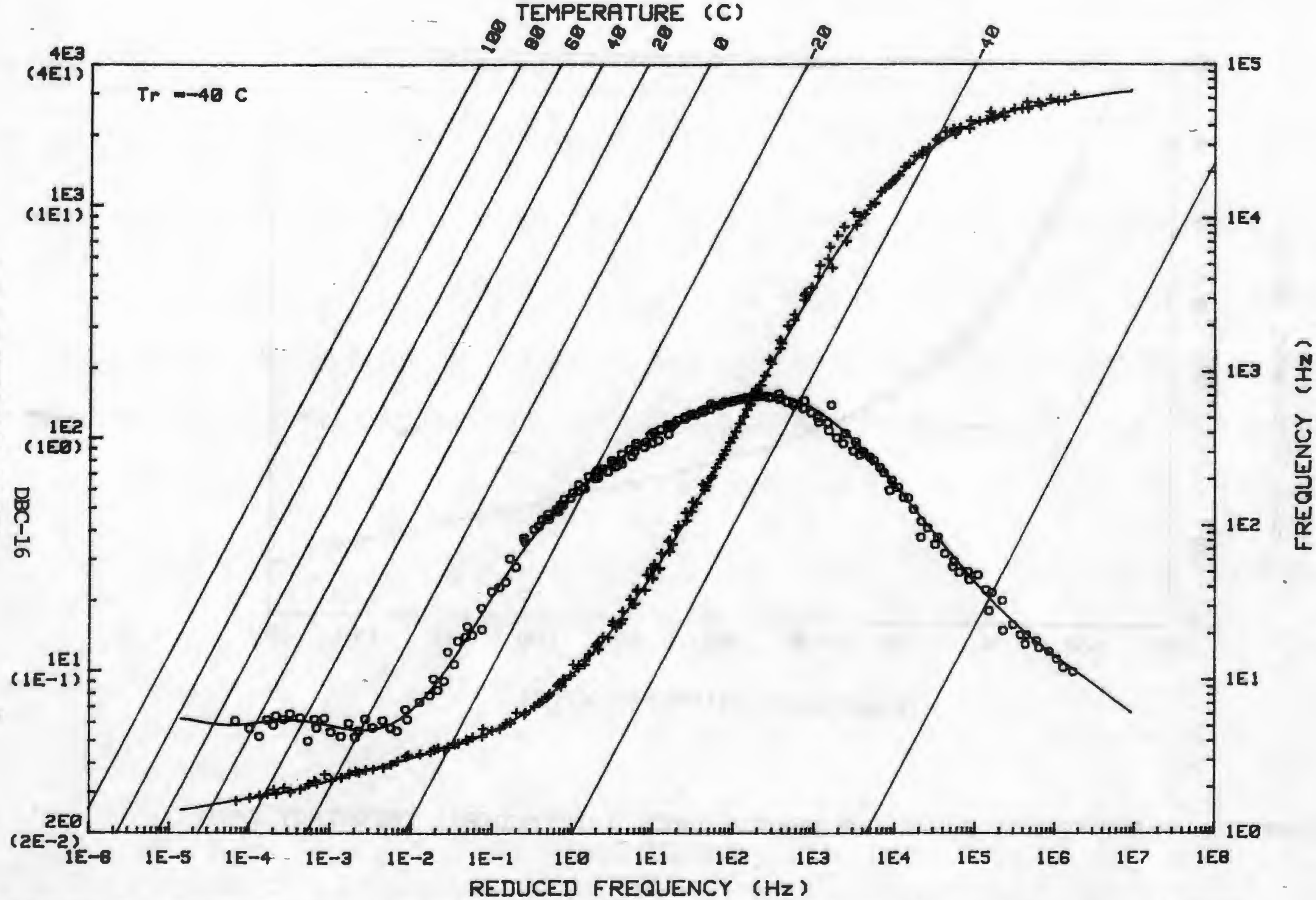

FIGURE 7 : INTERNATIONAL PLOT OF COMPLEX YOUNG'S MODULUS FOR POLYISOPRENE RUBBER (NARROW BAND, INTERMEDIATE FREQUENCY DATA)

DBC-16

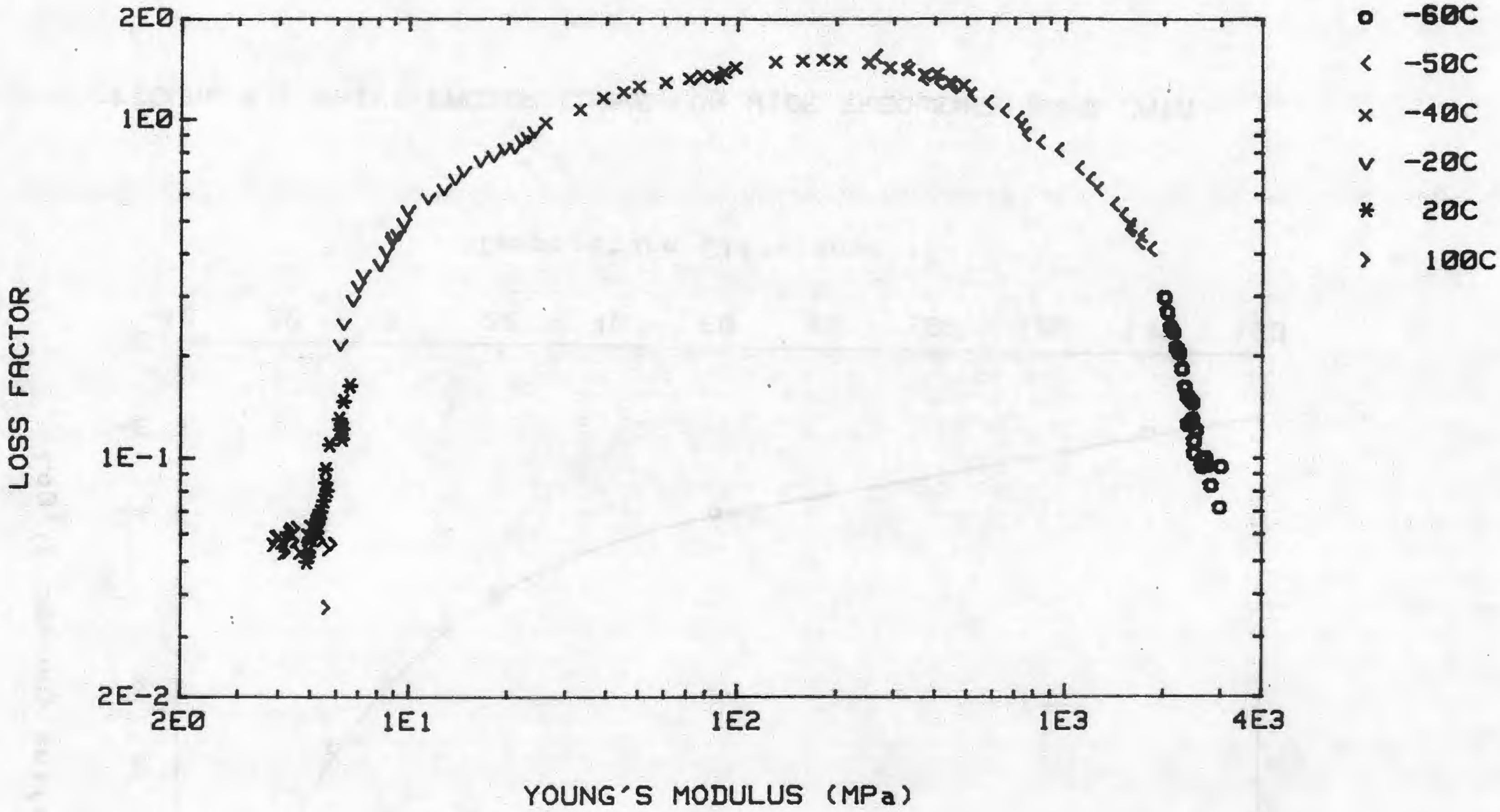

FIGURE 8 : LOSS FACTOR VS. MODULUS (WICKET PLOT) FOR POLYISOPRENE RUBBER
(WIDE FREQUENCY BAND DATA)

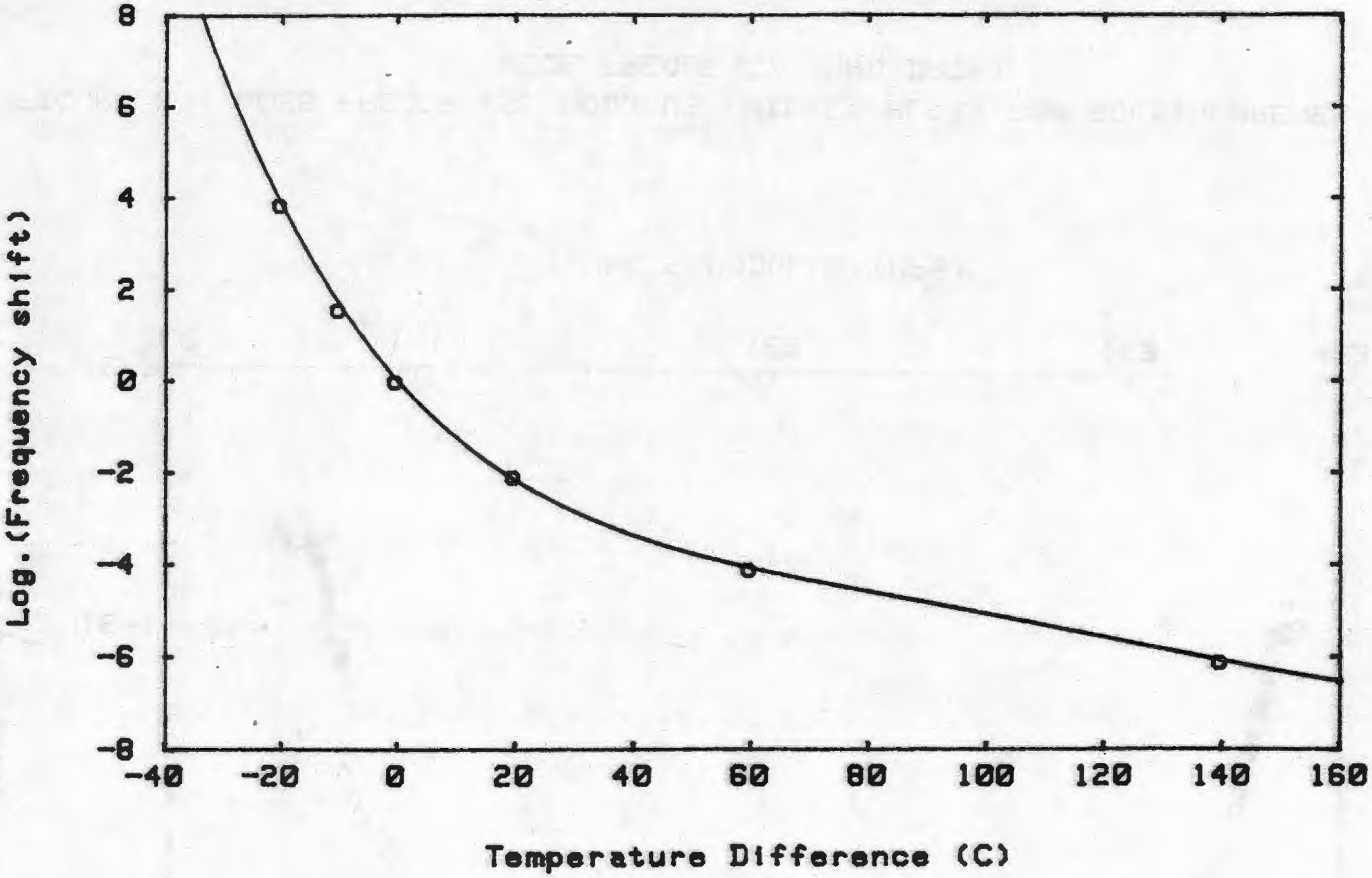

FIGURE 9 : SHIFT FACTOR CURVE FOR WIDE FREQUENCY BAND DATA

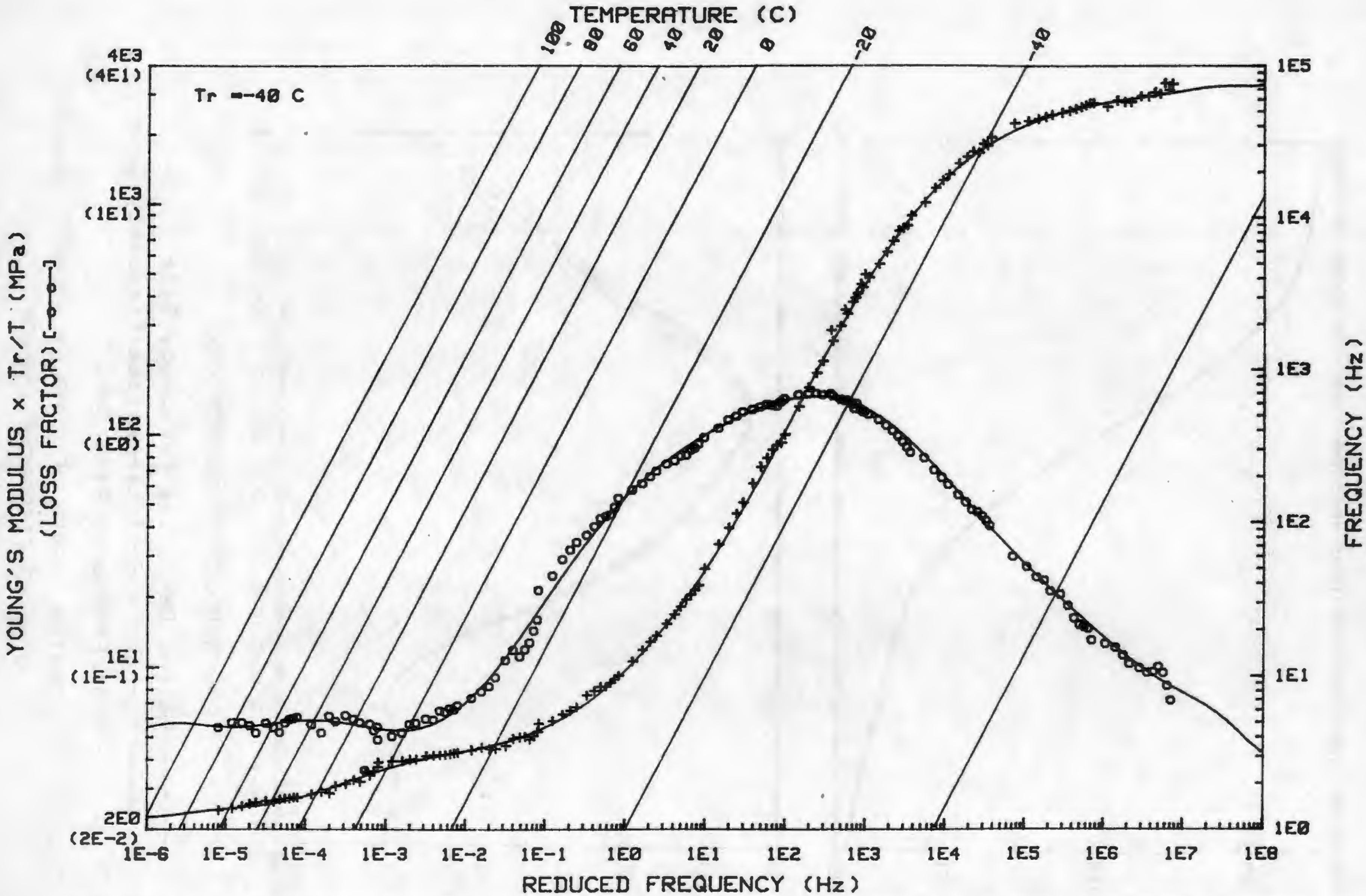

FIGURE 10 : INTERNATIONAL PLOT OF COMPLEX YOUNG'S MODULUS FOR POLYISOPRENE RUBBER (WIDE FREQUENCY BAND DATA)

DBC-19

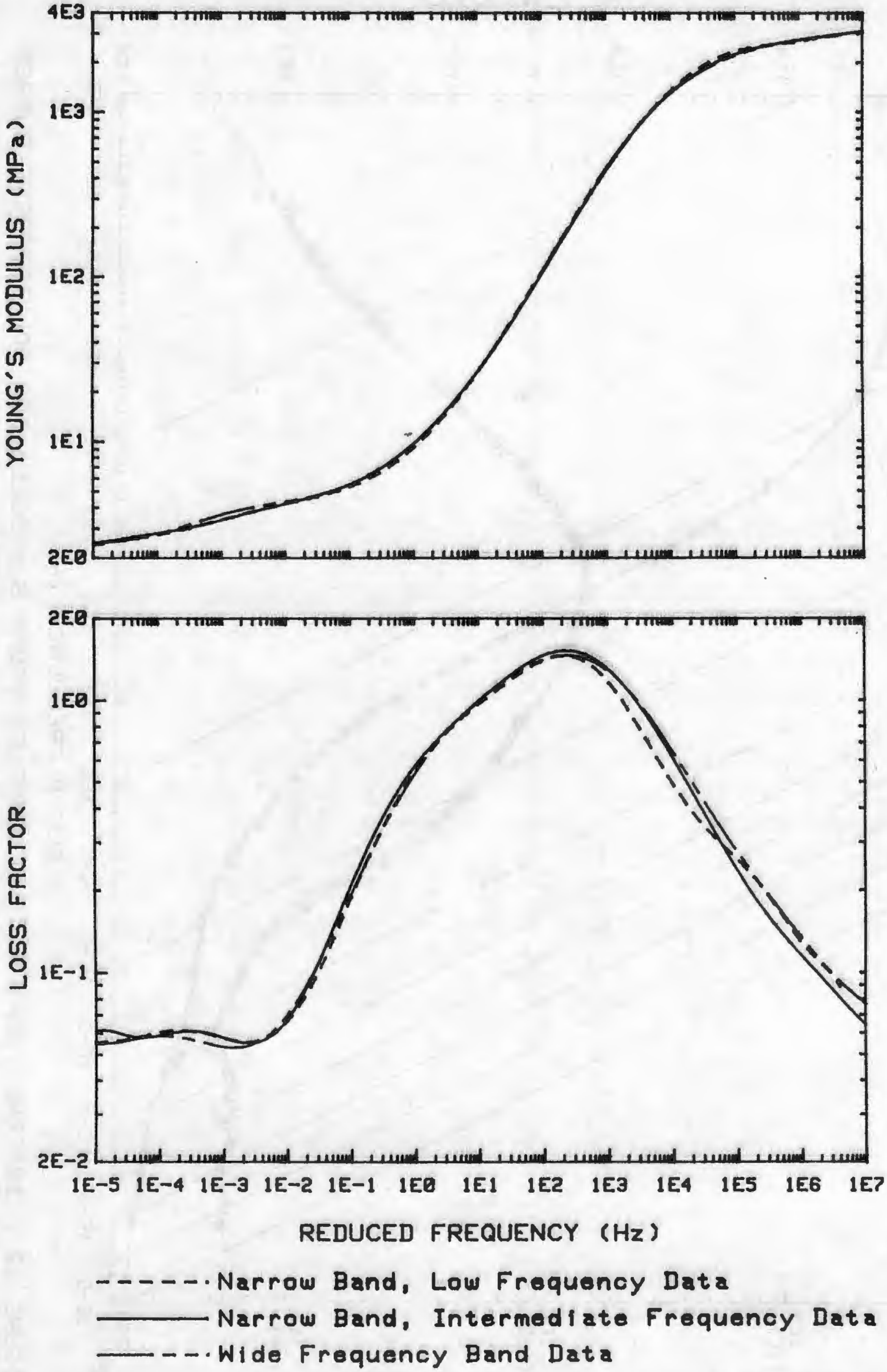

FIGURE 11 : MASTER CURVES OF COMPLEX YOUNG'S MODULUS FOR POLYISOPRENE RUBBER