CHEMICAL AND MECHANICAL PERFORMANCE OF FLEXIBLE MAGNETIC TAPE CONTAINING CHROMIUM DIOXIDE

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ABSTRACT

The development of a magnetic-recording tape using chromium dioxide suitable for use with the IBM 3480 Tape Drive was found to require careful optimization of the chemical and mechanical properties of the coating. This paper discusses the role of chromium dioxide in the oxidative and hydrolytic degradation of the polyester-polyurethane binders used in most flexible tape coatings, and the subsequent necessity for careful binder selection to eliminate (for all practical purposes) these degradative effects. In addition to the chemical behavior, the paper discusses the role of the interaction of the chromium dioxide with the binder necessary to obtain mechanical performance suitable for the 3480 tape drive. A relatively high glass-transition temperature and a sufficiently high modulus were found to be required to avoid changes in the frictional properties of the tape. The incorporation of a rough-textured back coat was found to control the change in the surface topography of the recording surface near the spool hub as a result of compressive forces.

oxidation-reduction reactions [5]. Early patents relevant to the use of chromium dioxide in the manufacture of magnetic tape claimed that a surface treatment or stabilization of the chromium-dioxide particles is required to stabilize the magnetic properties of the finished medium [6-9]. Little reference has been made, however, to the impact of this instability on the integrity of the coating and, particularly, to the chemistry of the binder resin.

The binders commonly used in the fabrication of flexible magnetic tape are elastomeric polyurethanes, polyvinyls, and combinations of these two major families of polymers. Other polymeric additives are often used to achieve the desired hardness, flexibility, toughness, and adhesion. Additional components such as lubricants, dispersants, and curative agents are also commonly added to the coating formulation. More details of state of the art in magnetic-tape formulations are available in several recent reviews [2-4].

Chemical analysis of tape samples containing chromium dioxide in various binder formulations, which had been exposed to thermal and humid aging, indicated changes in both the chemical functionality and the molecular weight of the polymer. An understanding of the mechanism of the interaction of the chromium dioxide with the binder and of the resulting changes in the coating performance was achieved through analysis of solutions and free films of the magnetic inks and from extracts obtained from coated tapes. Both free films and finished tapes were subjected to high temperature and humidity to monitor the effects of these aging environments.

Experimental

Coatings were made containing acicular chromium dioxide and γ-Fe_2O_3 dispersed into an organic solvent system containing the polymeric binder. Polyurethane binders constructed from 1,4-butanediol (as the hard-segment chain extender) and 4,4-diphenylmethane diisocyanate (MDI) with three soft-segments were used. Resin A contains an alcohol-terminated polyester as the soft-segment, whereas weights near 450 and 300 daltons were also used to standardize the column set. A third-order fit for the standards gave a 99.87% correlation coefficient with an estimated error of 6%.

(GPC results obtained for extracts of tapes are reported as amount of binder relative to lubricant and as the molecular weight (relative to styrene) for the peak maximum (M_p) of the molecular-weight distribution.)

Results and Discussion

The polyurethane elastomers used as binders for flexible-magnetic tape are composed of segments or blocks of chemically different units. These elastomeric polyurethanes contain a flexible, low-melting portion (soft-segment) connected to a more rigid, high-melting block (hard-segment). Typically, the soft-segment is composed of a repeating series of ester or ether-linked units of relatively short chain lengths. The polyester soft-segment blocks are formed by the reaction of a difunctional carboxylic acid with a difunctional alcohol such that the resulting polymer is terminated primarily with alcohol end-groups. The polyether soft-segments are constructed through a number of pathways usually involving the ring-opening condensation of cyclic ethers to give alcohol-terminated, ether-linked polymers, also of relatively short chain lengths.

The effect of changes in the length of the soft-segment is usually observed as an increase in the elasticity of the polyurethane made from the soft-segment polyol by the addition of difunctional isocyanate and a variety of chain extenders. The structure and chemical composition of the soft-segment (i.e., polyester or polyether) play a significant role in achieving solubility and performance properties, as well as dictating the polyurethane's degradation resistance [10-12]. The soft-segment portion of a polyester or polyether-polyurethane, therefore, plays a crucial role in defining the behavior of a given polyurethane binder for magnetic-tape applications.

of time. Hydrolysis of polyester-polyurethanes is well documented in the chemical literature [21-23] and is found to be the primary mechanism responsible for the deterioration of magnetic-tape performance after exposure to humid aging [17-19]. Hydrolysis involves the cleavage of the polyester chain of the soft-segments by the addition of water to the ester linkages. This reaction produces alcohol and acid functional fragments, with a rapid reduction in the molecular weight of the parent polymer. The significance of such binder degradation to tape performance arises from the relationship between the molecular weight of the polyurethane and its ability to produce a cohesive film when filled with magnetic particles. In addition, as stated in the literature, low-molecular-weight products of hydrolytic degradation can migrate to the surface of the tape and produce undesirable contamination of the recording head, with resultant changes in signal quality and frictional properties [17,18].

The motivation for the investigation of the impact of humid aging on tapes containing chromium dioxide is evident from the preceding discussion. Comparison of the susceptibility of the polyester and polyether-polyurethanes to humid aging when they are used as binders for chromium dioxide was therefore undertaken to assess the archival performance of such tapes. A comparison with conventional formulations using γ-Fe_2O_3 as the magnetic pigment was thus carried out to establish a bridge to previous investigations [19,20].

The results presented in Fig. 1 indicate that exposure to elevated temperature and humidity causes only slight or negligible changes in the molecular weight of the extractable binder for a tape containing γ-Fe_2O_3 as the magnetic pigment. These slight changes may be evidence of hydrolytic degradation of the binder or simply the result of adsorptive fractionation of the lower-molecular-weight portion of the original binder molecular-weight distribution. The lubricant peak in the GPC chromatogram, when used as an internal standard, suggests that the latter explanation is probably operative for the γ-Fe_2O_3 pigmented tape. The small increase in extractable binder with a slightly higher molecular weight indicates increased binder extraction after were found to migrate to the tape surface, leading to a substantial change in the frictional properties of the tape.

The unaged chromium dioxide tape performed satisfactorily on a conventional digital-recording tape drive. The same tape, after exposure to 42°C/20%RH for 29 days, still performed at an acceptable level, but an audible noise was detected by the operator during tape motion indicating some change in the ease of the unwinding of the tape from the reel. Another reel of the same tape, after exposure to 50°C/30%RH for 48 days, not only produced the audible noise during tape motion, but upon stop-start operations produced adhesion of the tape to the recording head and failure of the drive to move the tape on command. Additional aging at 52°C/30%RH for 82 days produced a severely degraded tape which could not be moved through the tape path nor readily unwound from the reel.

Examination of the recording head after running such degraded tape over the surface showed considerable tacky contamination, which was identified by mass spectrometry as consisting primarily of polyester fragments. Considerably fewer ~~less of the~~ mass spectral ions associated with the polyurethane hard-segments were observed in the mass spectra of the head contamination resulting from these degraded tape tests than were found present in either the undegraded binder or the extracts of the degraded tape. Presumably, the polyurethane fragments produced by hydrolytic degradation of the soft-segments, although present in the THF extracts of the tape, do not migrate to the tape surface as readily as the low-molecular-weight, polyester-rich, degradation fragments.

To stabilize the recording performance of chromium dioxide tapes, the mechanism of degradation, apparently catalyzed by the chromium dioxide, was investigated. Degradation of resin A when combined with chromium dioxide was found to be accelerated with higher temperature storage at a given humidity and at higher humidity for a given temperature. These observations are represented graphically in Figs. 3 and 4. No change in the molecular weight of the extracted binder, attributable to hydrolytic degradation, was noted for the tape stored under dry conditions. The decrease in the amount of chromium dioxide surface might preclude the effectiveness of a stabilizing additive in mitigating the chromium dioxide-catalyzed degradation of the polyester portions of the binder.

The substitution of a thermoplastic polyether-polyurethane was expected to offer a more fruitful resolution to the binder degradation associated with formulations based on chromium dioxide because their resistance to hydrolysis is well documented [21,25]. When a polyether-polyurethane (resin C) was substituted into the chromium dioxide formulation, however, quite the reverse was observed (Fig. 6). The amount of extractable binder of such low molecular weight prior to humid aging was unexpected. Examination of the GPC results obtained for the polyether-polyurethane before milling with the chromium dioxide indicates a narrow molecular-weight distribution with a peak maximum at 120,000 daltons. The extract obtained from the chromium dioxide-resin C tape before aging gives an extremely broad distribution, with its peak maximum near 8,200 daltons. The amount of extractable material, furthermore, was extremely high and actually exceeded the initial ratio of the binder relative to the lubricant, as measured by the differential-refractometer detector. This presumably results from a significant change in the refractive index of the fragments relative to the initial polymer.

The results obtained for the extract of the aged tape (Fig. 6) indicate that the humid aging increased the degradation of the polyether-polyurethane, but the significant observation to be derived is the apparent sensitivity of the polyether-polyurethane to the addition of chromium dioxide. Because the degradation is observed to occur without the environmental exposure, hydrolytic degradation does not appear to offer a reasonable explanation of the observed changes in the polyether in the presence of chromium dioxide. Oxidative chain scission of the polyether portion of the polymer occurs for these polyurethanes, giving rise to low-molecular-weight products and rapid deterioration of tape performance.

From the preceding discussion, it appears that stabilizers and polyether-polyurethanes might not offer a suitable solution to the apparent binder initial decrease in the molecular weight of the extractable binder to the fractionation of the polyester-polyurethane rather than to hydrolytic degradation.

Continued exposure to humid aging is then observed to give an increased amount of extractable binder with a decrease in molecular weight, as indicated in the GPC results presented in Fig. 7, curve D. This result is believed to be a consequence of the competition between water vapor and the urethane polar groups for the adsorption sites on the surface of the chromium dioxide particle. As the water successfully infiltrates the urethane-particle interface, disruption of previously insolubilized binder occurs, resulting in the marked increase in the extractable material. That this explanation is appropriate is further demonstrated by the results obtained after exposure to dry, thermal aging (Fig. 8).

The results presented in Fig. 8 were obtained by placing a chromium dioxide-resin B tape into a desiccator at 52°C for a period exceeding 60 days. The THF extracts of this sample produced the series of GPC chromatograms shown. The extract concentrations were adjusted to an equivalent amount and gave reproducible peak heights for the lubricant peak, thus permitting the superposition of the series of traces to give what is readily seen as a continuous fractionation. As dry thermal exposure is continued, the urethane insolubilization increases with the lower-molecular-weight fraction remaining soluble for the longest period. This presumably arises from the fact that more adsorption sites are present for the higher-molecular-weight polymer chains, which must subsequently be disrupted by the extraction solvent to effect solubilization. The shorter chains, having fewer polar sites capable of being tightly held on the particle surface, remain relatively soluble.

Comparison of the results obtained after dry, thermal aging with those obtained for a similar tape exposed to humid, thermal aging (Figs. 7 and 8) indicates that for the chromium dioxide -resin B formulation the primary action during the initial aging period is the insolubilization of the binder and an increase in the cohesive integrity of the coating, as shown by its in the characteristics of the tape surface also lead to changes in the recording characteristics, particularly to changes in amplitude, and to changes in frictional performance. The friction forces can become catastrophically high if the tape surface takes on the extreme smoothness of the base film under the interlayer pressures encountered when the tape is wound on a reel.

The frictional force in elastomeric systems consists of contributions from adhesion forces and hysteresis forces. The hysteresis term in viscoelastic (rubbery) materials is caused by a delayed recovery of the elastomer after indention by an asperity. The adhesion term is a result of the shearing of adhesive bonds formed at the interface in the region of the real area of contact. For magnetic-tape applications using polyurethane binders and operating under normal conditions, the adhesion component of friction is the most significant; the hysteresis component is negligible [28].

The adhesion component of friction (f_A) is defined as the product of the real area of contact (A_r) and the shear strength of adhesion (τ_a) between the surface contact,

$$f_A = A_r \tau_a \ . \tag{1}$$

The real area of contact for elastic contacts (true for light loads) is given by:

$$A_r \sim 3.2\ W/E_c \sqrt{\sigma_p/\beta_p} \tag{2}$$

and for plastic contacts,

$$A_r \sim W/H \ , \tag{3}$$

where W is the normal load, E_c is the reduced complex modulus, H is the indentation hardness, and σ_p and β_p are the composite standard deviation and

A number of tapes made by various tape manufactures were wound on the 3480 reels. Stress conditions for measuring changes related to creep deformation were selected to be 51°C and 60% relative humidity. Tapes were wound on the reels at 2.2 N of tension. Frictional measurements were then taken at selected intervals in the reel from the beginning of tape (BOT) to the end of tape (EOT) near the hub. Initially, measurements were made before the wound reels were subjected to the elevated temperature and humidity conditions, and then they were repeated after two-day stress periods. The measurements were made at 32°C and 87%RH. Tapes were conditioned to the test environment for 24 hours before being measured. Measurements were made on a model of the 3480 tape path. Because creep deformation caused by interlayer pressures is greatest at EOT, only EOT frictional measurements are reported. The friction measurements consisted of locating the end of tape at the head, relieving tension, and manually pulling the tape under initial tension of about 50 g provided by the drive motor. The breakaway force required to pull the tape was measured by a tensilometer. The wrap angle of the head was about 15°. The results are shown in Table 1. The effect of creep deformation caused by increasing interlayer pressures (decreasing radial reel position) for tape 2 after two days at 51°C and 60%RH is shown in Fig. 10.

Of the original tapes used in the experiment, the ones that showed the most stable frictional properties were 1, 5, 9, 10, 11, 15, 17, and 18. All of these eight tapes were back-coated, except for tapes 15 and 17. The binder systems of these two tapes contained a hard, organic polymer additive in significant ratios to the base polyurethane binder.

These results led to another experiment, in which identical coatings were applied to substrates of increasing smoothness. The same high-temperature and relative-humidity conditions and measurement intervals were used. Four substrates with varying smoothnesses were selected. By varying the surface smoothness, the friction could be varied from 55 to above 500 g. The experiments showed the important role that the surface finish of the substrate plays in influencing the forces developed by creep deformation.

corresponded reasonably with the temperature observed in the modulus curves obtained by DMA (Fig. 13). These observations suggested that at a temperature dictated by the thermomechanical properties of the coatings, the real area of contact between the tape asperities and the mating tape-drive surfaces can undergo a rapid increase under an applied stress. The stresses to which the asperities are exposed presumably determine the critical modulus at which compression of the asperities begins to become appreciable. After the critical temperature (modulus) is exceeded, the coefficient of friction is found to increase dramatically (Fig. 13).

As we noted earlier, the polyester-polyurethane binders have been found to exhibit considerable interaction with the chromium dioxide particles. Aside from the degradative effects already discussed, the polyurethane binders have been found to display a marked sensitivity to their thermal and environmental histories [30]. As shown in Fig. 14, when a free film of chromium dioxide formulation is exposed to humid, thermal aging, the modulus is found to increase significantly. This appears to be the result of thermally induced phase separation permitting greater wetting interactions with the chromium dioxide particle. Such effects from thermal aging are not unusual for filled coatings, especially for those involving strong polar-bond interactions [26,27].

The increase in the modulus of the coating after thermal aging suggests that a tape subjected to such aging without smoothening compressive forces should exhibit increased stability in its frictional performance as a result of the increased modulus. This was found to be the case for the experiment discussed before, in which a set of tapes was exposed to humid, thermal aging. One of the tapes was subjected to compressive stresses and was found to exhibit a marked increase in friction properties, especially near the reel hub, where the compressive forces are greatest. Another tape, which had been stored without any applied compressive force, was found to exhibit normal frictional behavior. When this thermally aged, low-friction tape was returned to the humid, aging environment, with the compressive forces renewed, the effect of the increased modulus during the first environmental cycle was marked. Only a hard, glassy polymers and the tailoring of a single polymer are effective in controlling the creep-deformation rate. The binder system and resultant modulus must be designed around the "use" temperature of the tape product. This ensures the stability of the tape surface and thus maintains its frictional characteristics without substantial risk of disturbing other performance requirements, such as abrasion resistance and head wear.

CONCLUSIONS

The magnetic particles of chromium dioxide possess inherently attractive properties for use in digital magnetic recording. The implementation of conventional polyurethane-binder technology with chromium dioxide formulations, however, was found to require more consideration of the particle-binder interactions than for other magnetic particle systems. The chemical and mechanical stabilities of the finished coating were found to require specific chemical properties in the binder for formulations based on chromium dioxide.

Oxidative reactions at the binder-chromium dioxide interface affecting the functionality of the polyurethane hard-segments were not found to produce a deleterious effect on tape performance. Hydrolytic degradation of the polyester soft-segments, however, was found to be accelerated by the interaction with the chromium dioxide particles and had an impact on the mechanical performance of the tape upon exposure to humid aging. A hydrolytically stable polyester-polyurethane was found to offer a significant improvement in the resistance of the coating to binder degradation.

Careful control of the mechanical properties and maintenance of a stable surface topography were found to be necessary for acceptable tape performance. The deformation of the coating under compressive stresses was found to require consideration of the modulus of the coating at the applied stress conditions and of the control of the surface topography and frictional properties of the coating.

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FIGURE CAPTIONS

1. Gel-permeation-chromatographic separation of γ-Fe_2O_3 with resin A: Curve A--THF solution of resin A with lubricant, without γ-Fe_2O_3; Curve B--THF extract of tape before humid aging; Curve C--THF extract of tape after humid aging for 21 days at 50°C/30%RH.

2. Gel-permeation-chromatographic separation of chromium dioxide with resin A: Curve A--THF solution of resin A with lubricant, without chromium dioxide; Curve B--THF extract of tape before humid aging; Curve C--THF extract of tape after humid aging for 21 days at 50°C/30%RH.

3. Change in the molecular weight of the extractable binder for the chromium dioxide-resin A tape; effect of temperature and humidity.

4. Change in the amount of extractable binder relative to the lubricant for the chromium dioxide-resin A tape; effect of temperature and humidity.

5. Gel-permeation-chromatographic separation of chromium dioxide-resin A tape containing carbodiimide stabilizer: Curve A--THF extract of unstabilized tape before humid aging; Curve B--THF extract of unstabilized tape after 21 days at 50°C/30%RH; Curve C--THF extract of stabilized tape before humid aging; Curve D--THF extract of stabilized tape after 21 days at 50°C/30%RH.

6. Gel-permeation-chromatographic separation of chromium dioxide-resin C tape: Curve A--polyether molecular weight distribution before addition to chromium dioxide; Curve B--THF extract of tape before humid aging; Curve C--THF extract of tape after 21 days at 50°C/30%RH.

7. Gel-permeation-chromatographic separation of chromium dioxide-resin B tape extracts: Curve A--THF extract of tape before thermal aging; Curve B--THF extract after three days at 52°C/60% RH; Curve C--21 days at 52°C/60% RH; Curve D--36 days at 52°C/60% RH; and Curve E--60 days at 52°C/60% RH.

Table 1. Static friction at end of tape for different tapes
(stressed at 51°C and 60%RH, in time)
tension (T_0) = 50 g, wrap angle = 15°

Tape No.	Friction (Δ tension) (g) with stress time						
	0 Days	2 Days	4 Days	6 Days	8 Days	18 Days	22 Days
1	55	55	55	55	55	55	55
2	55	>340	--	--	--	--	--
3	55	>340	--	--	--	--	--
4	55	>340	--	--	--	--	--
5	60	65	60	60	60	65	60
6	50	95	190	250	>340	--	--
7	55	>340	--	--	--	--	--
8	55	85	100	140	190	220	270
9	50	50	55	50	50	55	50
10	50	55	50	50	55	50	55
11	50	50	50	55	50	50	50
12	55	>340	--	--	--	--	--
13	60	65	120	130	160	170	180
14	55	160	220	>340	--	--	--
15	50	55	50	55	55	50	50
16	45	60	95	110	120	170	200
17	45	45	40	45	45	50	50
18	50	45	50	50	55	50	50
19	55	95	100	120	130	140	160
20	60	120	160	190	220	>340	--

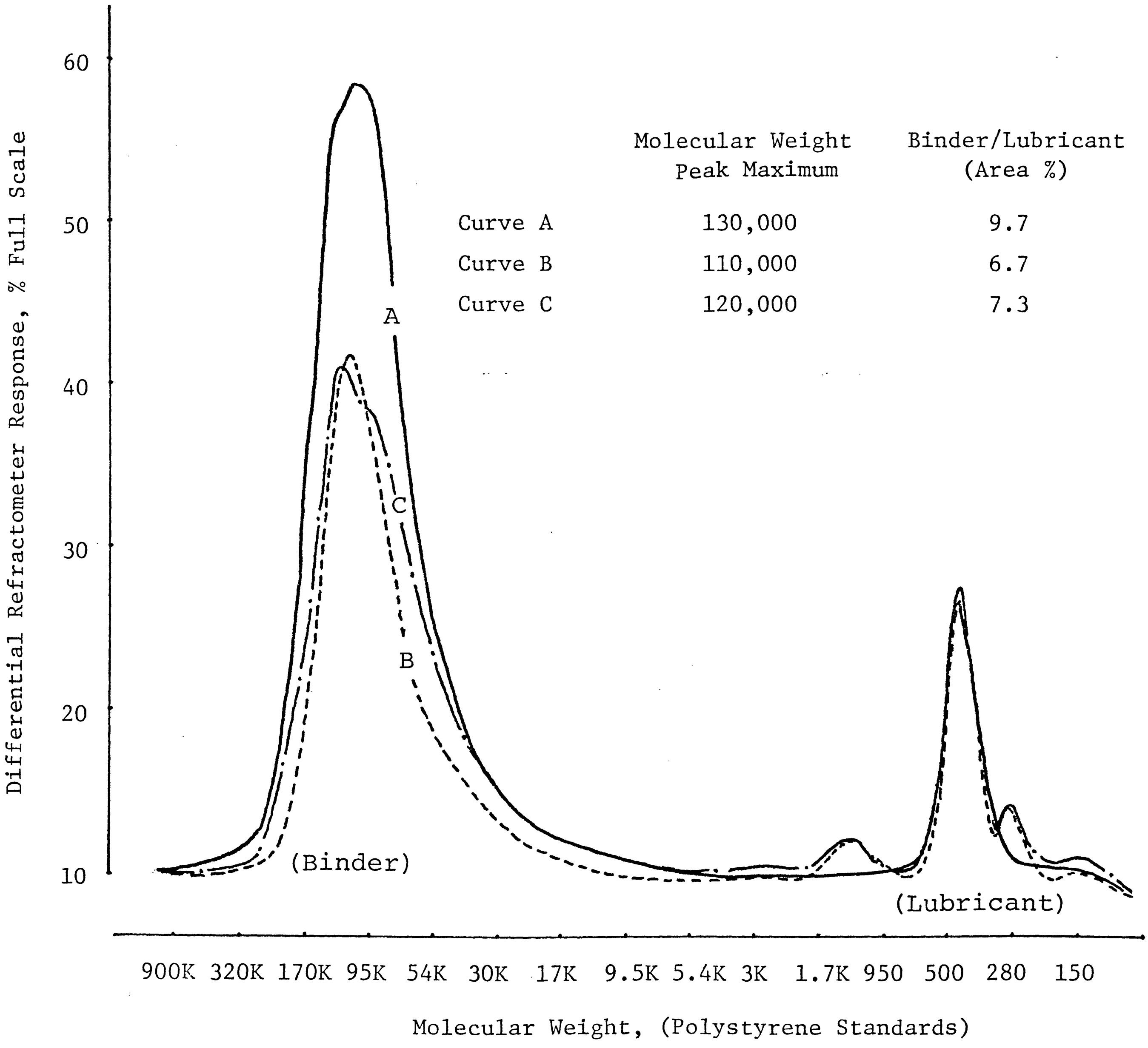

Differential Refractometer Response, % Full Scale
60
50
40
30
20
10
Molecular Weight Peak Maximum
Binder/Lubricant (Area %)
Curve A 130,000 9.7
Curve B 110,000 6.7
Curve C 120,000 7.3
A
B
C
(Binder)
(Lubricant)
900K
320K
170K
95K
54K
30K
17K
9.5K
5.4K
3K
1.7K
950
500
280
150
Molecular Weight, (Polystyrene Standards)

Figure 1.

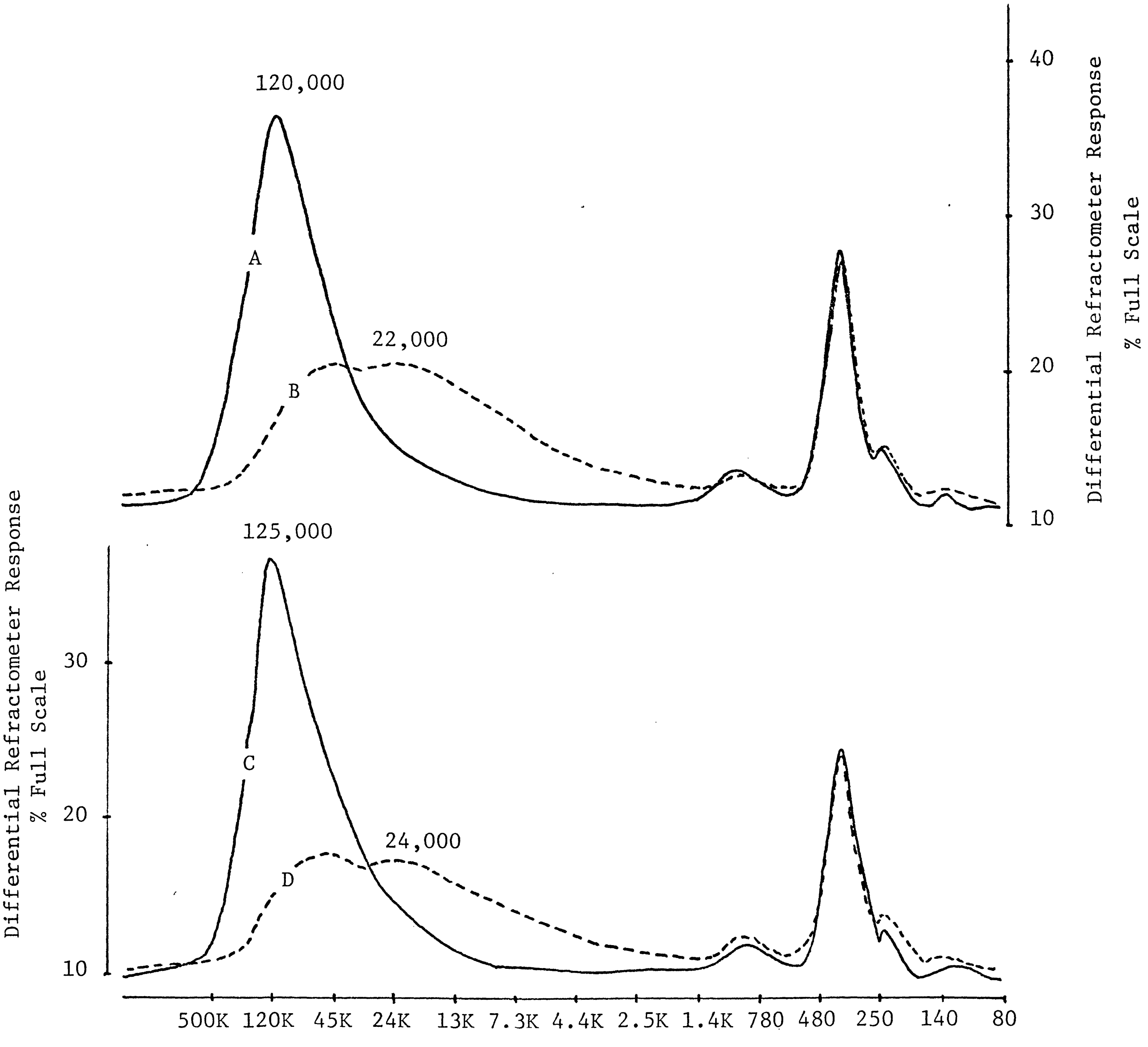

120,000
A
22,000
B
125,000
C
24,000
D
Differential Refractometer Response
% Full Scale
40
30
20
10
30
20
10
500K
120K
45K
24K
13K
7.3K
4.4K
2.5K
1.4K
780
480
250
140
80
Molecular Weight, (Polystyrene Standards)

Figure 5.

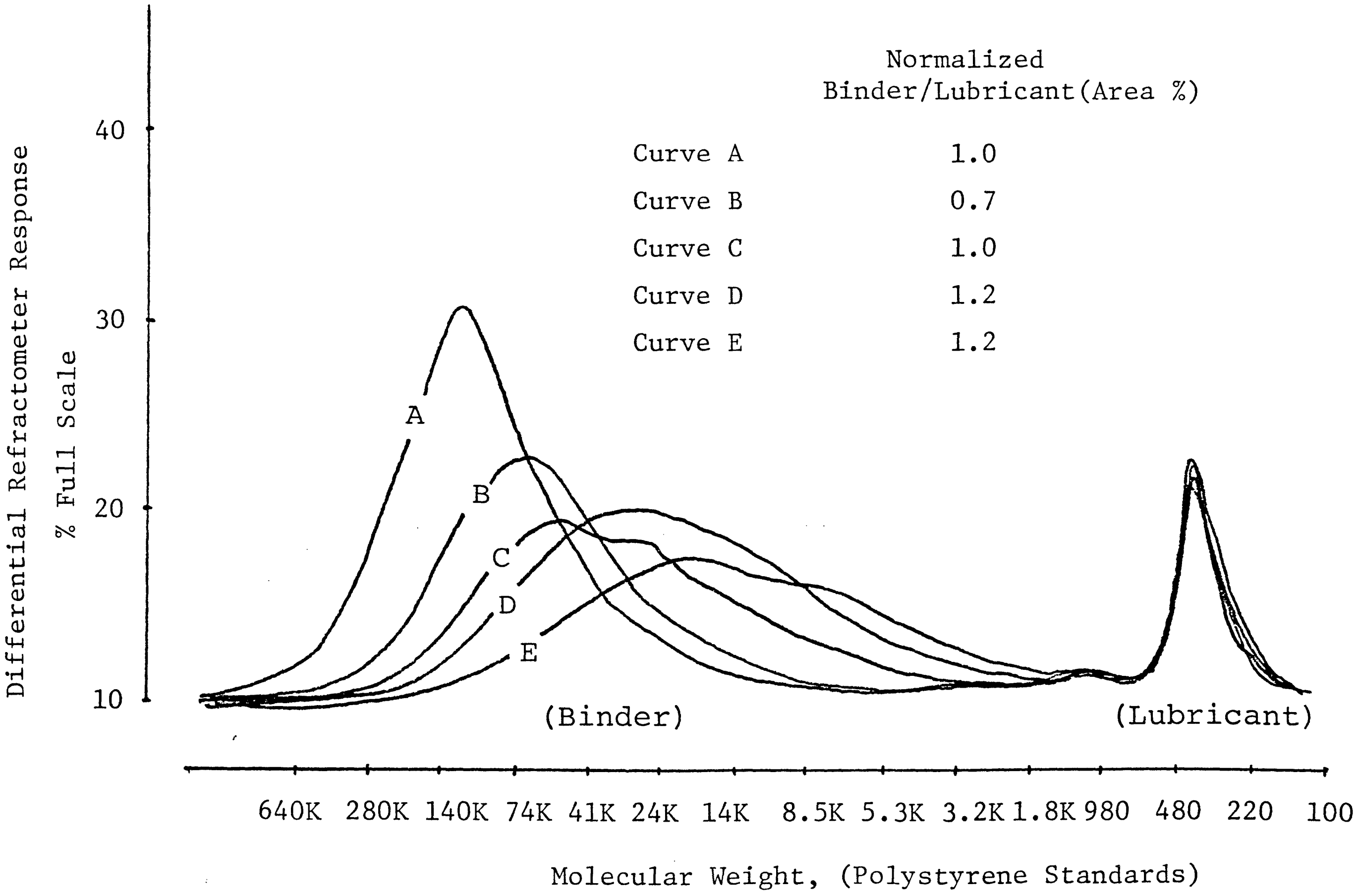
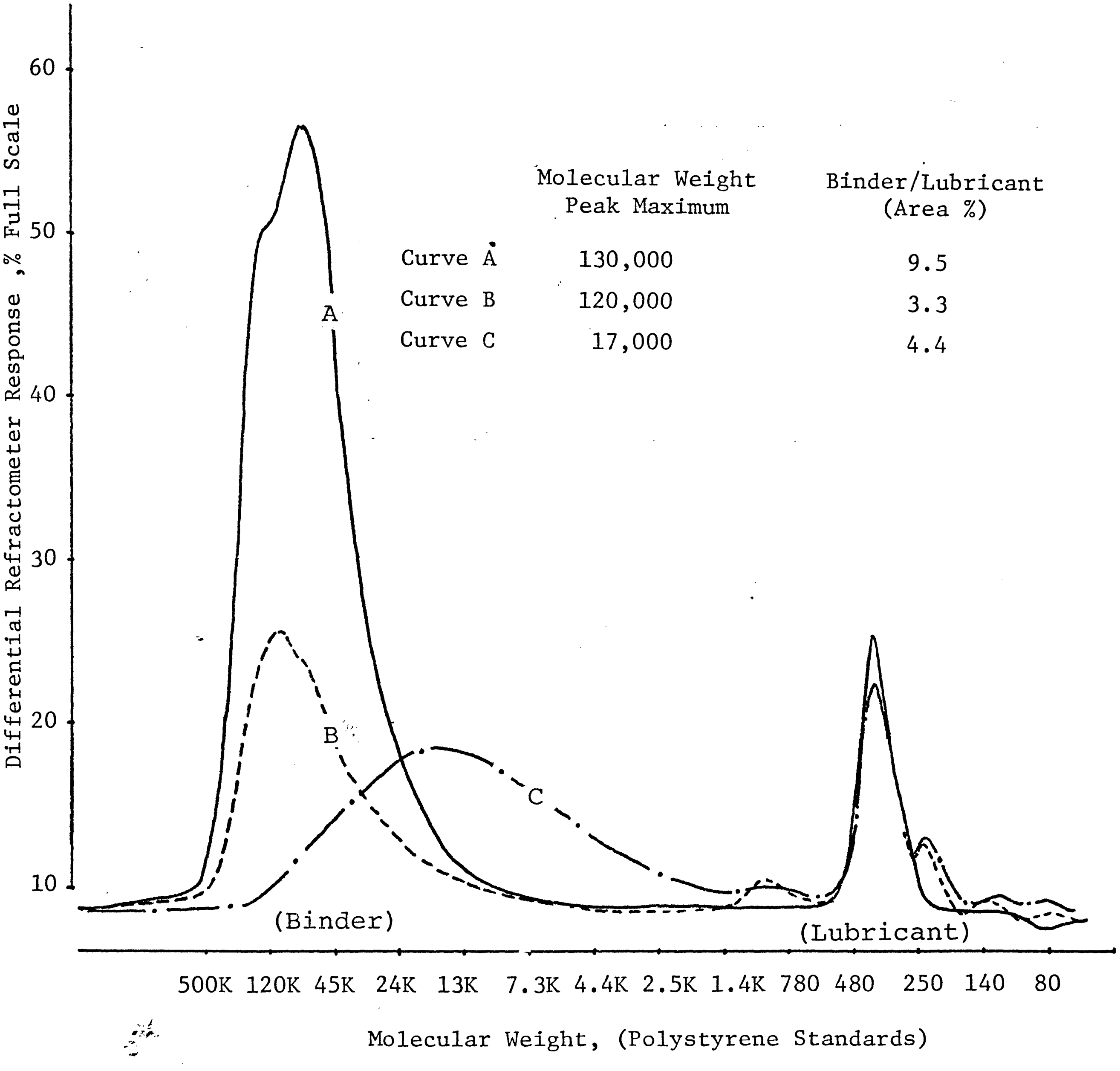

Normalized
Binder/Lubricant(Area %)
Curve A 1.0
Curve B 0.7
Curve C 1.0
Curve D 1.2
Curve E 1.2
Differential Refractometer Response
% Full Scale
40
30
20
10
A
B
C
D
E
(Binder)
(Lubricant)
640K 280K 140K 74K 41K 24K 14K 8.5K 5.3K 3.2K 1.8K 980 480 220 100
Molecular Weight, (Polystyrene Standards)

Figure 7.



	Molecular Weight Peak Maximum	Binder/Lubricant (Area %)
Curve A	130,000	9.5
Curve B	120,000	3.3
Curve C	17,000	4.4

Figure 2.

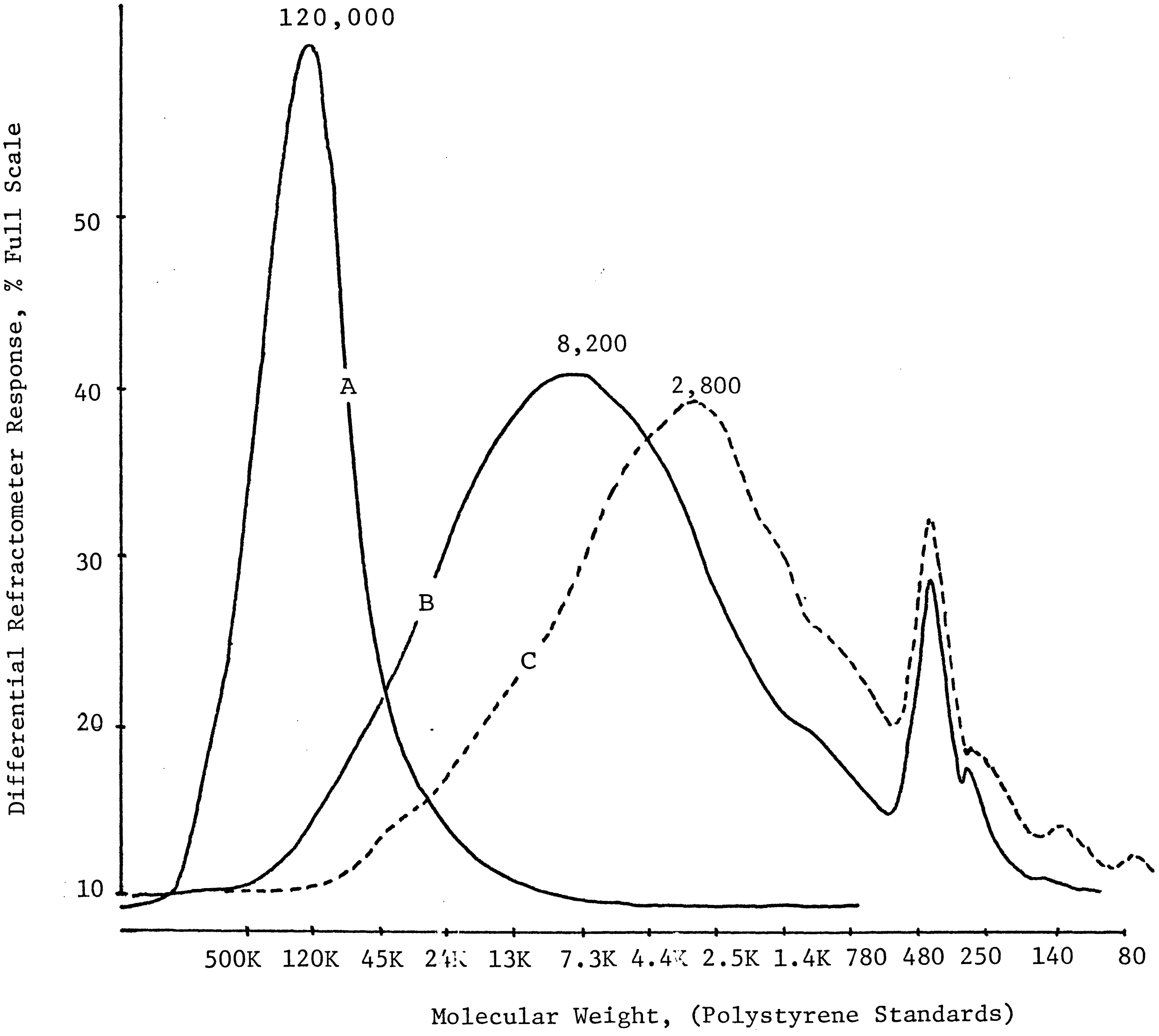

120,000
8,200
2,800
A
B
C
Differential Refractometer Response, % Full Scale
50
40
30
20
10
500K 120K 45K 24K 13K 7.3K 4.4K 2.5K 1.4K 780 480 250 140 80
Molecular Weight, (Polystyrene Standards)

Figure 6.

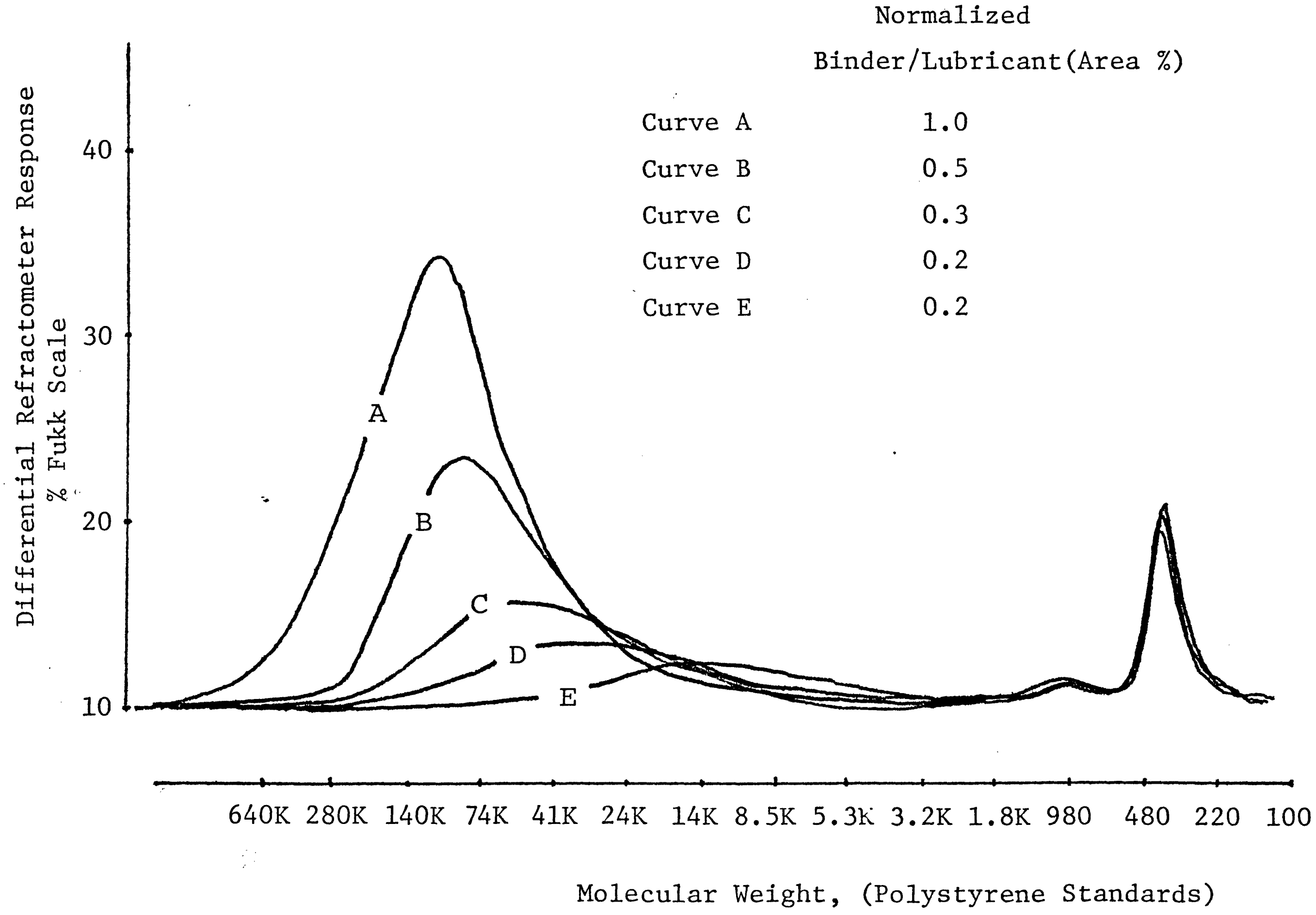

Normalized
Binder/Lubricant(Area %)
Curve A 1.0
Curve B 0.5
Curve C 0.3
Curve D 0.2
Curve E 0.2
Differential Refractometer Response
% Fukk Scale
40
30
20
10
A
B
C
D
E
640K
280K
140K
74K
41K
24K
14K
8.5K
5.3K
3.2K
1.8K
980
480
220
100
Molecular Weight, (Polystyrene Standards)

Figure 8.

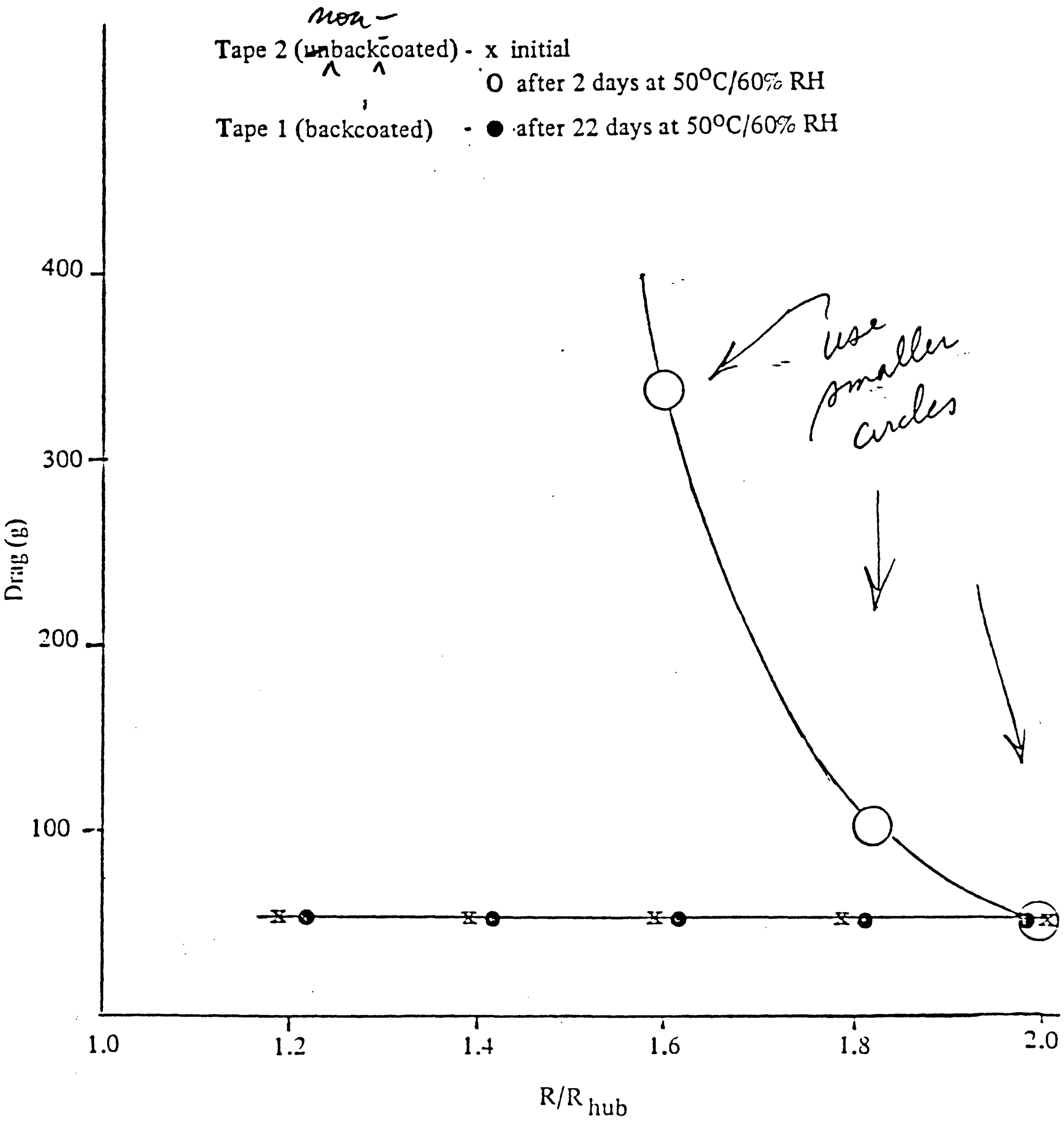

non-
Tape 2 (~~un~~backcoated) - x initial
O after 2 days at 50°C/60% RH
Tape 1 (backcoated) - ● after 22 days at 50°C/60% RH
use smaller circles
400
300
200
100
Drag (g)
1.0
1.2
1.4
1.6
1.8
2.0
R/R hub

Figure 10

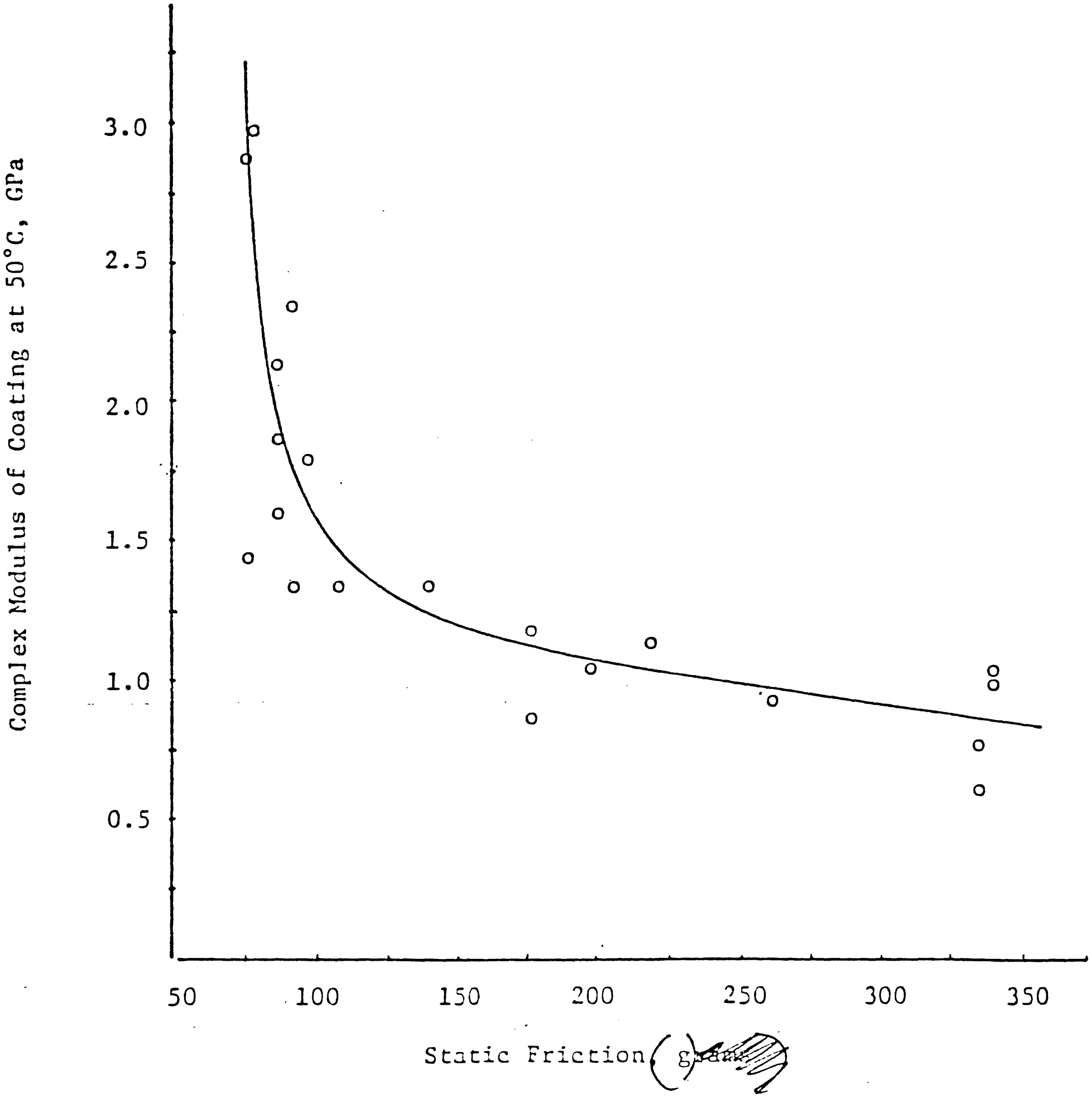

Complex Modulus of Coating at 50°C, GPa
3.0
2.5
2.0
1.5
1.0
0.5
50
100
150
200
250
300
350
Static Friction (g)

Figure 12

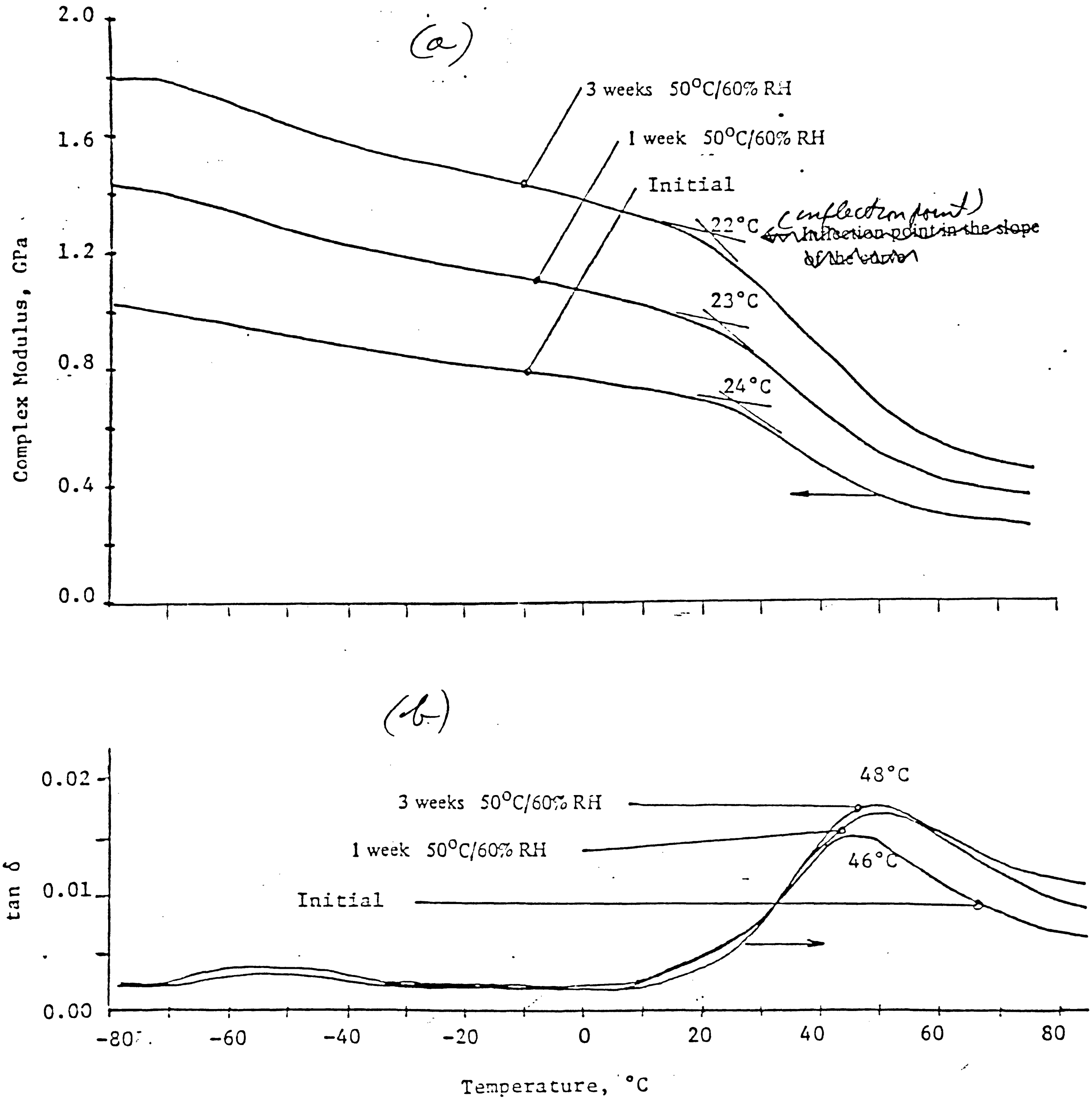
(a)
3 weeks 50°C/60% RH
1 week 50°C/60% RH
Initial
22°C
(inflection point)
~~Inflection point in the slope of the curve~~
23°C
24°C
Complex Modulus, GPa
2.0
1.6
1.2
0.8
0.4
0.0
(b)
48°C
3 weeks 50°C/60% RH
1 week 50°C/60% RH
46°C
Initial
tan δ
0.02
0.01
0.00
-80
-60
-40
-20
0
20
40
60
80
Temperature, °C

Figure 14