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Annealing of Poly(ethylene terephthalate)-Film-Based Magnetic Recording Media for Improved Dimensional Stability

PET-film-based magnetic recording media are subject to a number of dimensional instabilities, a major one being inplane shrinkage due to stress relaxation in the biaxially oriented substrate. Annealing the media allows stress relaxation without significantly degrading other properties and makes possible the use of higher track densities. Effects of annealing on mechanical properties, coefficients of thermal and hygroscopic expansion, and long-term dimensional stability of the media are described.

Introduction

Flexible magnetic storage media generally consist of a poly(ethylene terephthalate) film substrate which has been coated with a dispersion of γ-Fe_2O_3 particles in a polymer matrix. PET-film-based recording media are subject to several dimensional instabilities such as irreversible shrinkage of the media due to stress relaxation in the substrate film, reversible changes in media dimensions due to changes in temperature or relative humdity, and changes due to creep. The dimensional changes which result from these instabilities are not uniform but vary with direction in the plane of the substrate. Thus, a data track which is circular when written on a flexible disk becomes distorted over a period of time as the substrate dimensions change. This track distortion is a most important limitation to high density recording on PET-film-based disk media.

The biaxially oriented PET film used in flexible disk recording media is a semicrystalline material with very good toughness, flexibility, and mechanical properties. The film is produced commercially by extrusion of a polymer melt which is quenched and then oriented. It is oriented by drawing operations, in the machine direction and in the transverse direction, to produce balanced mechanical properties. Then it is heat set by restraining the film at its stretched dimension and heating briefly to 150–230°C. This operation increases the crystallinity of the film and reduces the tendency of the film to shrink. Shrinkage may be reduced further by heating the film at a temperature lower than the heat set temperature but with low stress on the film. Finally, the film is cooled and wound onto rolls [1].

A biaxially oriented PET film is metastable in two respects. First, the percent crystallinity of the film is much lower than the equilibrium crystalline content. Second, noncrystalline regions of the film contain frozen-in strains which tend to relax and allow the film to contract [2]. The drawing operation causes orientation of both crystalline and noncrystalline regions of the film. As crystallites are formed in an oriented film, they hold in place many molecular chains which are already oriented. These chains are not free to retract even though the film temperature is above the glass transition temperature of the bulk material [3]. Time- and temperature-dependent relaxation of these ordered noncrystalline regions in PET film has been considered to be the major cause of shrinkage of the film.

Orientations of both crystalline and noncrystalline regions in PET film determine the properties of the film and the anisotropy in the properties [2, 4]. Various workers, whose work has been reviewed by Heffelfinger and Knox [1], have used wide angle x-ray diffraction to study crystalline orientation in PET films. When the film is drawn uniaxially during its manufacture, the C axis of the polymer crystal, which is the polymer chain axis, tends to align along the stretching direction [5]. When the film is then drawn in the transverse direction, oriented regions in the film fan out from the machine direction toward the transverse direction, with the degree of transverse orientation increasing with distance from the center of the web, and the C axes distribute themselves accordingly. The variation in orientation across a film web is the primary cause of anisotropy of film properties.

PET films must be oriented to develop the desired strength, stiffness, and toughness. To improve the dimensional stability of the films, various annealing treatments have been used which reduce internal stresses and increase crystallinity [6]. Annealing of PET films by a continuous process is difficult because any stress applied to the film must be kept low to minimize induced stresses in the film and because a relatively long time at the elevated temperature may be required if the film is to be annealed without adversely affecting its flatness and mechanical properties.

Magnetic recording media require substrates as dimensionally stable as possible. If the substrate film is annealed in sheet form, zero stress on the film is possible, and the annealing time can be as long as desired. Since the application of the magnetic coating is best done as a continuous process, it is desirable that the sheets be annealed after coating. Annealing sheets of media also relieves stresses induced in the substrate during the coating and curing operations.

This report describes annealing treatments and their effects on the properties of sheets of PET-film-based magnetic recording media. For flexible disk media, the properties of interest include static properties such as mechanical properties, coefficients of thermal and hygroscopic expansion, and long-term dimensional stability. Dynamic properties such as creep and fatigue properties are also of interest in some applications, but in this report only the static properties of media are discussed. Because of variations in degree of crystallinity and orientation, the properties of PET film vary with location in a roll of film and also from one roll of film to another. Therefore, in this study these variables had to be controlled as much as possible.

Since PET films are commercially important materials, the properties of the films have been determined by a number of workers. Mechanical properties have been published by film manufacturers [7–9] but these give properties only in machine and transverse directions. Several researchers [10, 11] have determined mechanical properties at various in-plane orientations. They found that PET films exhibited significant anisotropy in modulus, ultimate strength, and ultimate elongation, but that yield stress was essentially isotropic in these films.

Chu and Smith [4] determined mechanical properties of 36-μm (1.42-mil) PET film annealed at temperatures from 95 to 180°C. They found that annealing tended to decrease the modulus of the material, with higher annealing temperatures leading to lower modulus when annealing time is held constant. However, they reported properties only from 0 to 90° with respect to the machine direction of the film and did not consider all in-plane directions.

Values for coefficient of linear thermal expansion of PET films have been published by film manufacturers [7–9] and by Barrall and Logan [2]. According to data of the latter, thermal expansion is strongly dependent on in-plane direction in these films. Annealing at temperatures to 180°C did not appear to have a predictable effect on thermal expansion. Values for coefficient of hygroscopic expansion have also been published by film manufacturers [7–9], but not for various orientations.

Greenberg et al. [12] used rotating disk testers to study shrinkage, creep, and expansion coefficients of 36-μm (1.42-mil) PET disks, some uncoated and some with a magnetic coating. They found that the maximum shrinkage of a PET film disk at 72°C occurred along the machine direction of the film. Thermal and hygroscopic expansion coefficients were measured on one coated, annealed disk. The thermal expansion coefficient was strongly anisotropic, but the hygroscopic expansion coefficient was essentially isotropic.

In the work reported here, static tests were used to measure expansion coefficients and shrinkage. The static tests are much more precise than dynamic tests for the determination of dimensional changes in PET-film-based media.

Experimental procedures

Two recording media were studied. The 1 1/2-mil medium consisted of a 36-μm (1.42-mil) substrate of du Pont Mylar [13], Type A-PB (Precision Base) PET film which was coated on both sides with a 2.5-μm (100-μin.) coating of γ-Fe_2O_3 particles dispersed in a polymer binder. The 3-mil medium was composed of a 76-μm (3-mil) substrate of Mylar A-PB coated on both sides with a 2.5-μm-thick coating. Both media were produced by IBM.

Sheets of the media were cut from rolls and annealed in a circulating air oven controlled to ±1°C. During the annealing process, the sheets were supported horizontally

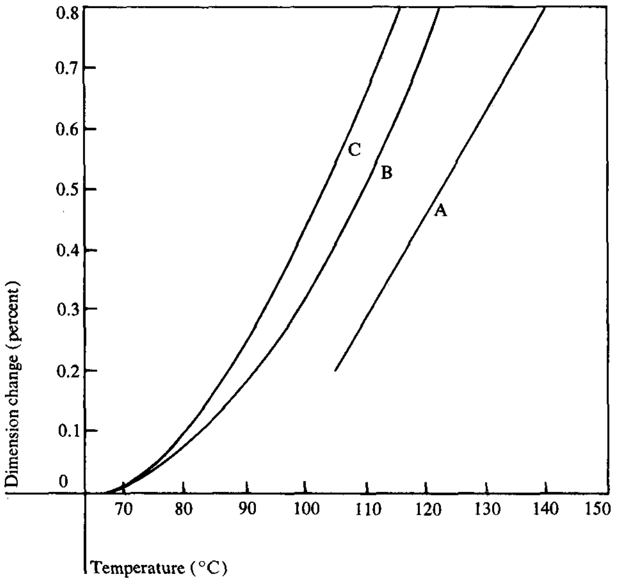

Figure 1 Shrinkage of 1 1/2-mil medium in machine direction after annealing at given temperature: curve A, 10 min; curve B, 2 h; curve C, 8 h.

on thin aluminum plates separated by spacers. The aluminum plates were preheated to the annealing temperature when the annealing time was one hour or less, but for longer annealing times the plates were not preheated. When annealing was completed, the plates and sheets of the media were allowed to cool to room temperature on a bench top. Annealing in this manner produced little change in the flatness of the 1 1/2-mil medium and improved flatness in the 3-mil medium.

Tensile modulus, yield stress, ultimate strength, and ultimate elongation of annealed and unannealed media were measured using die-cut dogbone-shaped specimens (ASTM D638, type IV). Specimen thickness was measured with a mechanical comparator to ±0.25 μm (±10 μin.). Tests were conducted using an Instron testing machine with a one-inch extensometer for measurement of strain in the specimens. A preload of about 7 MPa (1000 psi) was required to straighten the specimens before the extensometer was applied. The strain rates used were 0.05 min^{-1} for measurement of Young's modulus and 0.5 min^{-1} for measurement of yield and ultimate properties. At least three specimens were tested at each direction. Tests were conducted at approximately 23°C except where noted otherwise. It is important to note that, since PET films are viscoelastic, the property value measured in a given test is dependent upon the strain rate used.

Coefficients of thermal expansion of media were determined with a du Pont 942 Thermomechanical Analyzer, using the technique of Barrall and Logan [2]. At least three measurements were made along each direction. The specimens used were 3 mm (0.118 in.) wide with 7-mm (0.275-in.) lengths between the chucks. Tests were run at a heating rate of 5°C per minute.

Coefficients of hygroscopic expansion were measured using specimens 21 cm (8 1/4 in.) square. Three specimens of each material were used. Targets were lightly scribed into the magnetic coating of the media and dimensional changes were measured with a coordinate measuring microscope which reads to ±0.25 μm (10 μin.). Specimens were conditioned in an environmental chamber to 8 ± 1% relative humidity at 22 ± 1°C for at least 24 hours. When the chamber was opened, the specimens were pressed between glass plates and the distances between targets were measured. The specimens were then conditioned to 80 ± 1% relative humidity at 22 ± 1°C for at least 24 hours. After pressing between glass plates, the specimen dimensions were remeasured. The specimens were then returned to 8% relative humidity and measured again. Coefficients of hygroscopic expansion were calculated from the average of the two measurements made by increasing and then by decreasing the relative humidity.

Tests of long-term dimensional stability were conducted using the 21-cm (8 1/4-in.)-square specimens with scribed targets. The specimens were conditioned to 22 ± 1°C and 50 ± 1% relative humidity before measurement of dimensions and they were pressed between glass plates during measurement. After the initial measurements, the specimens were placed on aluminum plates separated by spacers and put into a circulating air oven at 60 ± 1°C. Periodically, the specimens were removed from the oven, returned to the equilibrium temperature and relative humidity, and then measured. Tests were run in triplicate and dimensional changes at 0°, 45°, 90° and 135° with respect to the substrate machine direction were recorded.

Results and discussion

Annealing

Figure 1 shows plots of shrinkage versus temperature at constant annealing time for the 1 1/2-mil medium. Shrinkage of the medium at temperatures below the glass transition temperature (T_g) of the PET substrate, about 69°C, was negligible with the times used. At temperatures above the T_g of the PET, the shrinkage of the medium was approximately linear with temperature when annealing time was held constant. Figure 2 shows shrinkage data on the 3-mil medium at constant time. The 3-mil material exhibited shrinkage behavior which was similar to that of the 1-1/2-mil medium.

When temperature was held constant and annealing time was varied, the 3-mil medium shrank as shown in Fig. 3. At temperatures above the T_g of the substrate, the medium shrank rapidly at first and then at a much slower rate. Within the range of times and temperatures used in this study, the medium continued to shrink with increasing annealing time. However, since the majority of the shrinkage took place rapidly at a given temperature, annealing for a relatively short time should significantly reduce stresses in the medium and thereby improve dimensional stability. For example, annealing the 3-mil medium two hours at 107°C (225°F) shrank the material about 0.5%. Longer annealing times produced more shrinkage but most occurred during the first two hours.

Variation of shrinkage during annealing with in-plane direction was also determined. Although the shrinkage varies significantly with direction, the shrinkage-versus-time plots for various directions are similar in shape to those in Fig. 3. Shrinkages in the machine direction were the maximum shrinkages or were near the maximum shrinkages when compared to the shrinkages in the other directions.

The amount of shrinkage which results from annealing PET-film-based media depends upon time and temperature. To determine which combination of time and temperature should be used, the properties of the media after annealing must be considered. For an efficient operation, the annealing temperature to be used should be as high as possible and the annealing time as short as possible. In magnetic recording media, annealing must be accomplished without significantly affecting the flatness or roughness of the material.

The flatness of sheets of the media was measured using a surface plate and feeler gauges to locate the largest waves on both sides of specimens 21 cm (8 1/4 in.) square. Before annealing, the 1 1/2-mil medium had no measurable waviness but the 3-mil medium contained waves over 1 mm (0.039 in.) in height. Some of the waviness in the 3-mil material was a curvature resulting from storing the material in roll form. Annealing the 1 1/2-mil medium at 107°C formed waves no higher than 0.08 mm (0.003 in.) and annealing at 120°C formed waves no higher than 0.25 mm (0.010 in.). In the 3-mil medium, annealing reduced the height of the larger waves so that the annealed material was flatter than the unannealed. Maximum wave height was 0.15 mm (0.006 in.) after annealing at 107°C and less than 1 mm (0.039 in.) after annealing at 120°C. At annealing temperatures above 120°C, the waviness of the annealed media was significantly greater than the waviness of the unannealed materials. It should be noted that, in some cases, annealing led to an increase in the number of small waves present, especially in the 1 1/2-mil medium annealed at the higher temperatures, but the number of waves present in a given area was not measured. Measurements of surface roughness using a profilometer showed the annealed media to have essentially the same surface roughness as the unannealed media.

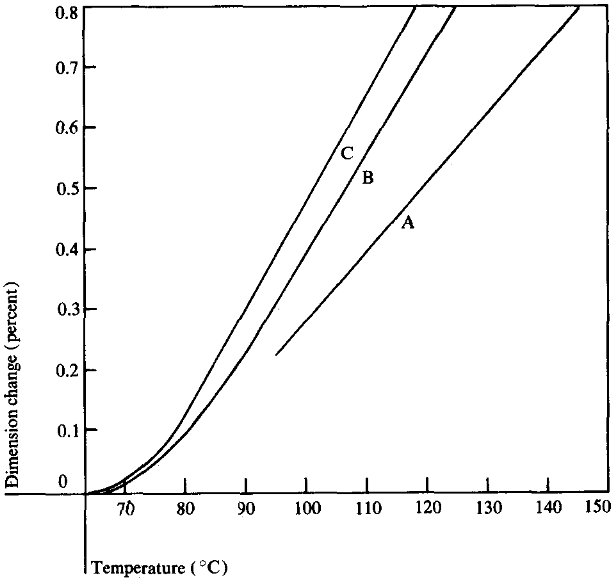

Figure 2 Shrinkage of 3-mil medium in machine direction after annealing at given temperature: curve A, 10 min; curve B, 2 h; curve C, 8 h.

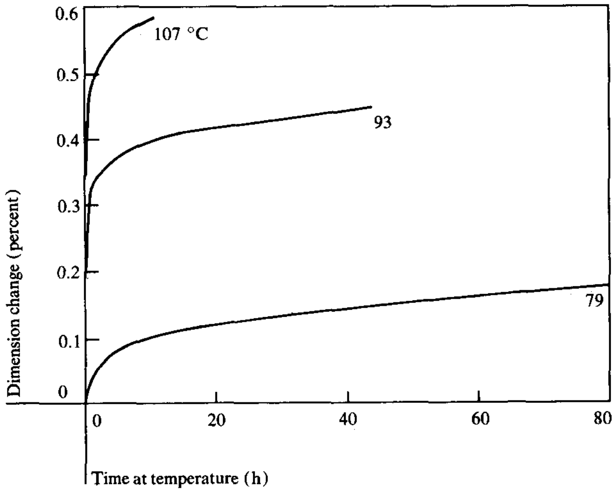

Figure 3 Shrinkage of 3-mil medium at constant temperature.

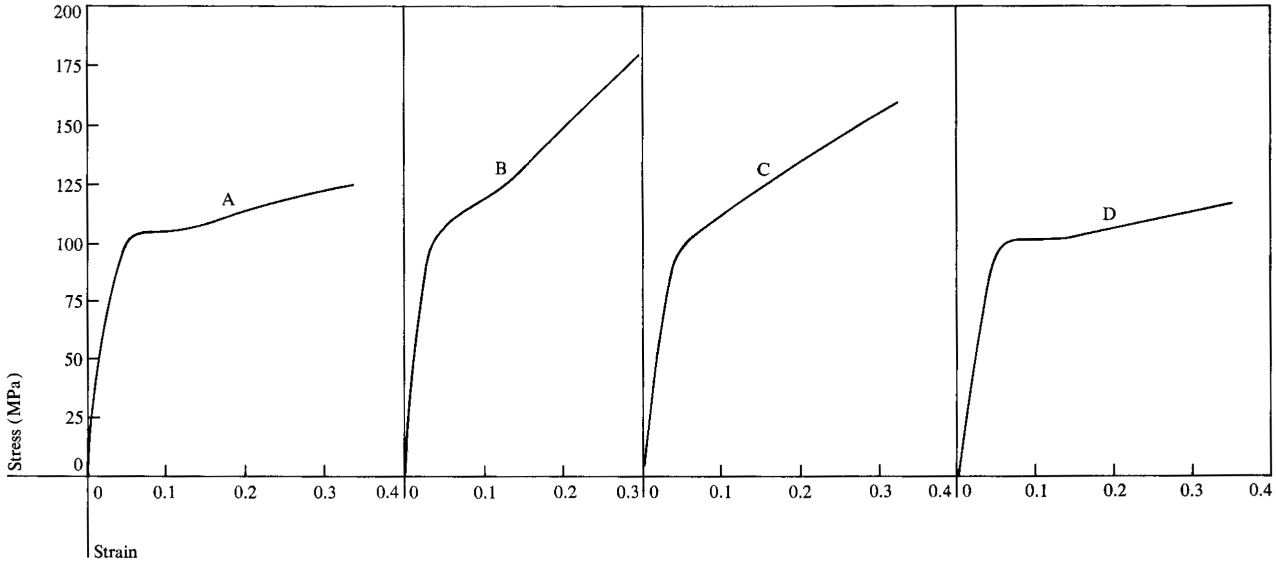

Figure 4 Stress-strain curves for unannealed 1 1/2-mil medium: curve A, machine direction; curve B, principal optical axis (70° to MD); curve C, transverse direction; curve D, transverse optical axis (160° to MD).

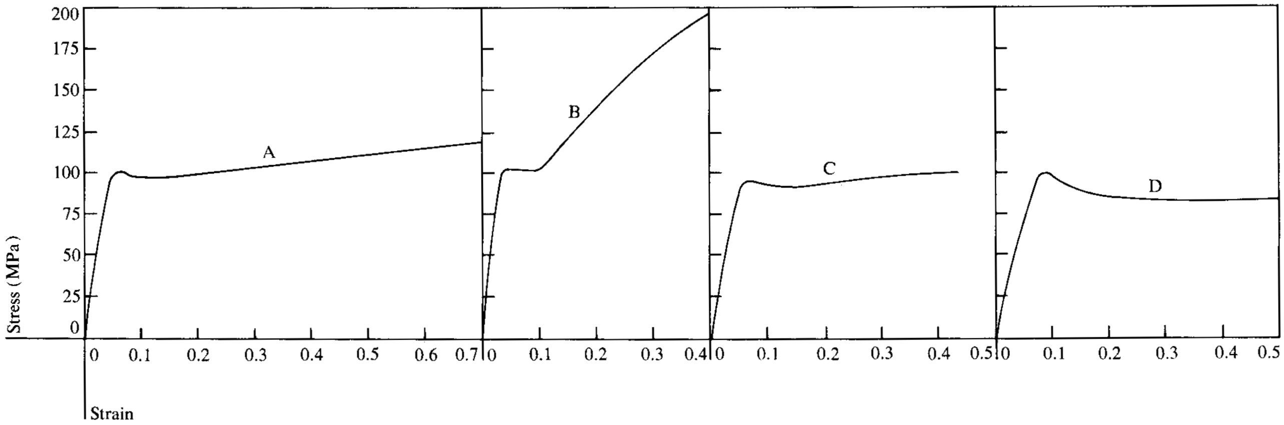

Figure 5 Stress-strain curves for unannealed 3-mil medium: curve A, machine direction; curve B, principal optical axis (45° to MD); curve C, transverse direction; curve D, transverse optical axis (135° to MD).

Because annealing of media is a stress relaxation process which produces shrinkage of the in-plane dimensions, it is of interest to determine whether there is a concomitant increase in the thickness of the media. Measurements of change in media thickness due to annealing showed that annealing at temperatures of 107 to 120°C produced thickness increases of approximately 1%.

This study shows that 1 1/2- and 3-mil PET-based magnetic recording media can be annealed in sheet form under zero stress. Annealing temperatures to 120°C have been used to produce about 0.5% shrinkage of the media without significantly affecting flatness or roughness. This annealing caused no measurable changes in magnetic properties of the media.

Mechanical properties

The mechanical properties of PET-film-based magnetic recording media are primarily determined by the properties of the PET film substrate. In a PET film, orientations of both crystalline and noncrystalline regions contribute to the mechanical properties of the film. Orientation di-

Table 1 Mechanical properties of media before and after annealing, machine direction.

Material	*Young's modulus* [GPa (10^5 psi)]	*Yield stress* [MPa (10^3 psi)]	*Ultimate tensile strength* [MPa (10^3 psi)]	*Ultimate elongation* *(%)*
1 1/2-mil medium, not annealed	4.38 (6.35)	105 (15.3)	122 (17.7)	32
1 1/2-mil medium, annealed 2 h at 107°C	4.30 (6.23)	104 (15.1)	119 (17.3)	30
1 1/2-mil medium, annealed 10 min at 120°C	3.98 (5.77)	101 (14.7)	124 (18.0)	40
3-mil medium, not annealed	4.44 (6.44)	103 (14.9)	119 (17.3)	69
3-mil medium, annealed 2 h at 107°C	4.42 (6.41)	100 (14.5)	114 (16.5)	65
3-mil medium, annealed 10 min at 120°C	4.45 (6.46)	98 (14.2)	115 (16.7)	66

rection can be determined by examining the film under crossed polaroids to obtain the optical axes of the film. The principal optical axis indicates the principal orientation direction in the film and the transverse optical axis is oriented at 90° to the principal optical axis. In the 1 1/2-mil medium, the principal optical axis of the substrate was found to be 66° to 70° from the machine direction, moving across the web, and in the 3-mil medium, the principal optical axis was 44° to 47° from the machine direction.

Figure 4 shows typical stress-strain curves for the unannealed 1 1/2-mil medium along the machine and transverse directions and along the optical axes of the substrate. Figure 5 shows typical curves for the 3-mil medium. The curves show that the yield stress is essentially independent of direction. After yielding, the stress-strain behavior of the media is strongly dependent upon the material orientation direction and not upon the machine direction. Similar observations were made by Chu and Smith [4] in their study of PET film properties, although they only measured properties from 0° to 90° with respect to the machine direction of the film.

Stress-strain curves of media annealed at temperatures to 120°C were very similar to the curves for the unannealed media. Table 1 shows data for mechanical properties of the media before and after annealing. Although properties were measured at 15° increments from 0° to 180° with respect to the machine direction of the media, only the properties along the machine direction are shown. Changes in properties with annealing were small in all cases and the amount of change was about the same for all in-plane directions.

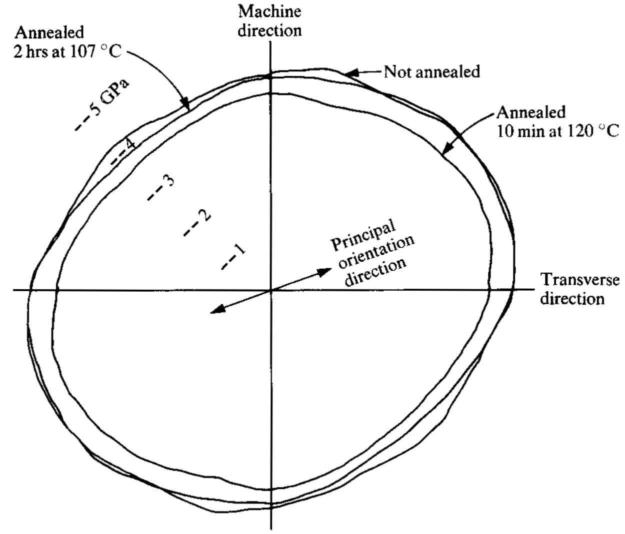

Figure 6 Young's modulus versus direction, 1 1/2-mil medium.

Figures 6 and 7 are plots using polar coordinates of Young's modulus versus direction in unannealed and an-

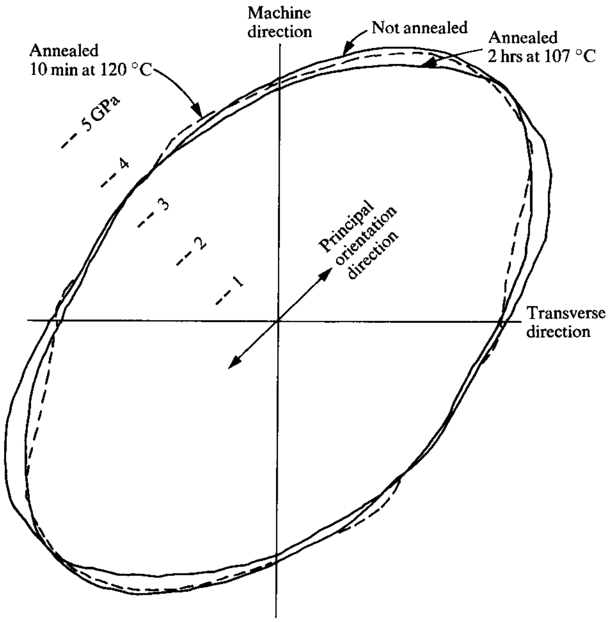

Figure 7 Young's modulus versus direction, 3-mil medium.

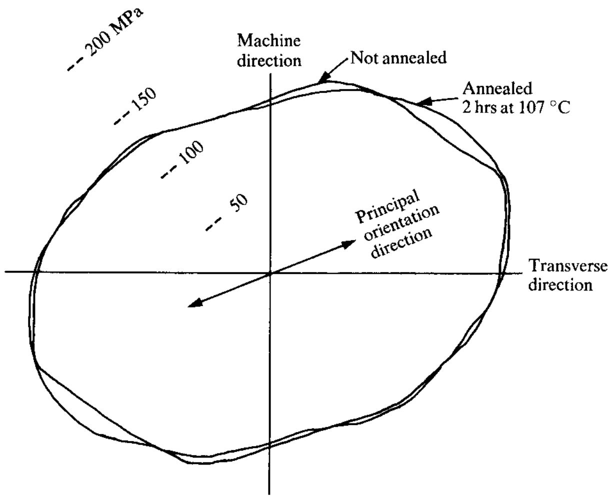

Figure 8 Ultimate tensile strength versus direction, 1 1/2-mil medium.

Table 2 Coefficients of linear thermal expansion of media, $10^{-6}(°C)^{-1}$.

Material	*Machine direction*	*Transverse direction*	*Principal optical axis*	*Transverse optical axis*
1 1/2-mil medium, not annealed	16.2	10.0	7.8	18.7
1 1/2-mil medium, annealed 2 h at 107°C	14.6	11.3	9.6	17.9
1 1/2-mil medium annealed 10 min at 120°C	14.8	12.6	11.0	18.2
3-mil medium not annealed	22.7	25.1	14.2	33.4
3-mil medium, annealed 2 h at 107°C	22.9	20.7	14.0	29.1
3-mil medium, annealed 10 min at 120°C	22.3	26.4	15.4	32.8

nealed media. Although the modulus was dependent upon direction in all of the plots, annealing the media resulted in essentially no change in the shape of the plots. In both of these materials, the maximum modulus was observed along the principal orientation direction and the minimum modulus was transverse to this direction. Annealing tends to reduce the modulus of oriented PET films by allowing relaxation of ordered noncrystalline regions in the films. In this study, relatively mild annealing conditions were used, so the modulus values for annealed materials were only slightly less than values for unannealed materials.

The annealing treatments used also produced little change in the yield or ultimate properties of the media. Figure 8 is a polar plot of ultimate tensile strength of the 1 1/2-mil medium before and after annealing. Chu and Smith [4] found that ultimate strength and elongation of biaxially oriented PET film increased with annealing, possibly due to higher crystallinity. However, the relatively mild annealing conditions used in this study caused only small changes in ultimate strength and elongation.

It is apparent that, in PET-film-based recording media, mechanical properties other than yield stress depend upon orientation in the substrate film. In both unannealed and annealed materials, maximum modulus and ultimate strength and minimum elongation were found along the principal orientation direction. Minimum modulus and ultimate strength and maximum ultimate elongation were found transverse to the principal orientation direction.

Coefficient of linear thermal expansion

Coefficient of linear thermal expansion was calculated from the slope of the best straight line drawn through the desired temperature range, in this case 25 to 50°C, on a TMA plot of probe displacement versus temperature. Experimental values for coefficient of linear thermal expansion of unannealed and annealed media are given in Table 2. Plots of coefficient of thermal expansion versus direc-

Table 3 Coefficient of hygroscopic expansion, $10^{-6}(\%RH)^{-1}$.

Material	*Machine direction*	*Transverse direction*	*Principal optical axis*	*Transverse optical axis*
1 1/2-mil medium, not annealed	7.3	6.9	6.3	8.0
1 1/2-mil medium, annealed 2 h at 107°C	8.4	8.0	7.1	9.6
1 1/2-mil medium, annealed 10 min at 120°C	9.6	8.8	7.5	9.7
3-mil medium, not annealed	6.8	7.9	6.5	9.3
3-mil medium, annealed 2 h at 107°C	8.5	9.1	7.8	9.2
3-mil medium, annealed 10 min at 120°C	10.5	12.5	10.3	11.5

tion in the 1 1/2-mil medium are shown in Fig. 9. Coefficient of thermal expansion was strongly anisotropic and dependent upon orientation in both unannealed and annealed media. The minimum coefficient of thermal expansion was observed in the principal orientation direction and the maximum coefficient was transverse to this direction. Annealing increased the lower values of thermal expansion coefficient in most cases and did not significantly affect the higher values, so the annealed media were somewhat less anisotropic in thermal expansion than the unannealed media.

Coefficient of hygroscopic expansion

Experimental data for coefficient of hygroscopic expansion are shown in Table 3. Figure 10 shows plots of coefficient of hygroscopic expansion versus direction in the 1 1/2-mil medium. The media exhibited significantly less anisotropy in hygroscopic expansion than in thermal expansion. Annealing the media increased coefficients of hygroscopic expansion. The annealing conditions used here allowed relaxation of oriented noncrystalline regions of the films, and this relaxation may allow the materials to absorb more moisture.

Long-term dimensional stability

Annealing reduces but does not eliminate internal stresses in oriented PET films. After annealing, PET-film-based media continue to shrink slightly due to continued relaxation of strained noncrystalline regions in the media substrates. The amount of shrinkage observed in an-

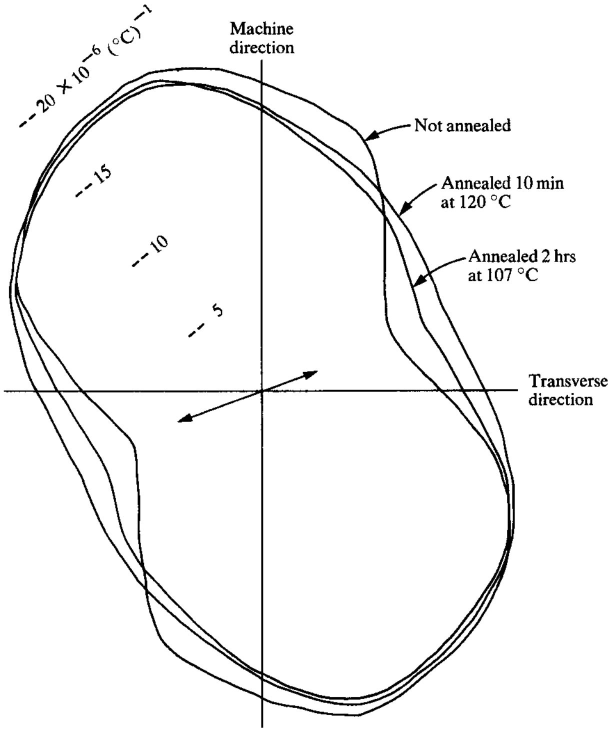

Figure 9 Coefficient of linear thermal expansion versus direction, unannealed 1 1/2-mil medium.

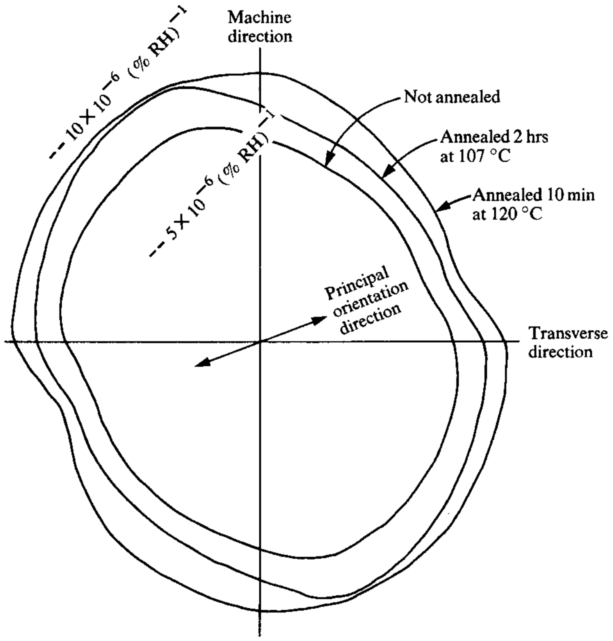

Figure 10 Coefficient of hygroscopic expansion versus direction, unannealed 1 1/2-mil medium.

Table 4 Shrinkage of media at 60°C.

Material	*Hours at 60°C*	*Total shrinkage (%)*			
		0°[a]	*45°*	*90°*	*135°*
1 1/2-mil medium, not annealed	168 2016	0.035 0.061	0.030 0.062	0.021 0.039	0.029 0.057
1 1/2-mil medium, annealed 2 h at 107°C	168 2016	0.010 0.009	0.009 0.013	0.007 0.011	0.010 0.012
1 1/2-mil medium annealed 10 min at 120°C	168 2016	0.007 0.019	0.015 0.021	0.016 0.022	0.015 0.020
3-mil medium not annealed	168 2016	0.029 0.074	0.026 0.065	0.014 0.027	0.024 0.044
3-mil medium, annealed 2 h at 107°C	168 2016	0.004 0.010	0.018 0.017	0.011 0.016	0.012 0.008
3-mil medium, annealed 10 min at 120°C	168 2016	0.008 0.010	0.018 0.017	0.013 0.018	0.011 0.009

[a] With respect to machine direction of substrate.

nealed materials depends upon time and temperature and upon the residual stresses in the film after annealing. Tests of long-term dimensional stability of media were conducted at 60°C and zero stress. Data for shrinkage of unannealed and annealed media are given in Table 4. The data for the unannealed 3-mil medium show that the maximum shrinkage was obtained in the machine direction and the minimum shrinkage occurred in the transverse direction. The 1 1/2-mil medium showed some tendency to shrink in this manner but the correlation of shrinkage with direction was not as good as for the 3-mil material.

The annealed media shrank much less than the unannealed media during long-term exposure to 60°C, as would be expected. However, the variation in shrinkage with direction in the annealed materials appears unpredictable from these data. After annealing, shrinkage does not appear to relate to substrate machine direction or to material orientation.

Conclusions

Mechanical properties of PET-film-based magnetic recording media were strongly dependent upon orientation in the film substrate. Maximum values of ultimate strength and Young's modulus occurred along the principal orientation direction in these materials. Maximum ultimate elongation was 90° from the principal orientation direction. Thermal expansion was also related to orientation with the minimum coefficient of thermal expansion found along the principal orientation direction and the maximum coefficient at 90° to this direction. In unannealed media, the maximum shrinkage was found along the machine direction of the substrate and the minimum shrinkage was found along the transverse direction of the substrate.

Annealing the media under the relatively mild conditions used in this study did not significantly change the ultimate properties or the coefficients of thermal expansion of the media. Young's modulus values were reduced slightly by annealing, and coefficients of hygroscopic expansion were increased by annealing. Annealing greatly improved the long-term dimensional stability of PET-film-based media.

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