Precision Dielectric Measurements of Nonpolar Polymers in the Millimeter Wavelength Range

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Abstract —**Complex refractive index and complex dielectric permittivity and loss tangent data for polyethylene, polypropylene, poly-4 methylpentene-1 (TPX), and polytetrafluorethylene (Teflon) are presented over the frequency range 60–300 GHz. All of these *nonpolar polymers* are extremely low-loss materials. As in the microwave region, polyethylene demonstrates exceptionally low-loss characteristics in the entire millimeter-wave region. *Polar polymers such as nylon and plexiglass exhibit much higher absorption (nearly an order of magnitude higher) loss compared to nonpolar polymers.***

I. INTRODUCTION

POLYMERS ARE EXCELLENT candidates as low-loss dielectric materials for millimeter-wave applications such as lenses, windows, and dielectric waveguides. These materials are very easy to fabricate to any shape and size. The most commonly used high molecular weight organic polymers are polyethylene terephthalate (known as Myler) polystyrene, polymethylmethacrylate (plexiglass), polyethylene, polypropylene, poly-4-methylpentene-1 (known as TPX), and polytetrafluorethylene. The absorption coefficient or the loss tangent of mylar, polystyrene, and plexiglass are very high in the submillimeter [1]–[3] region which forbids their use as window or lens material. However, thin films of these are in extensive use as beam splitters in the far- and mid-infrared spectral region. Nonpolar polymers are relatively low loss and become potential candidates for use as lenses, windows, and waveguide materials.

The most common nonpolar polymers are the last four in the list above. The TPX is transparent in the visible wavelength region and is superior to the others in its hardness and resistance to deformation by heat [4]. Its refractive index in the submillimeter-wave region (~1.46) also matches closely with its refractive index in the visible region (~1.43). This avoids the realignment of an instrument equipped with TPX lenses or windows once it is aligned using visible radiation.

PTFE (Teflon) is relatively softer but it is inert to all known organic liquids [5], [6]. It is very low loss in the radio frequency region but the absorption increases with frequency. A very strong absorption band [5], [7] appears in the submillimeter-wave range at 6 THz, which precludes its routine use beyond 4.50 THz. The microwave loss of unsintered PTFE exhibited an extremely low value (40 μrad).

Polyethylene and polypropylene have been familiar to radio and microwave frequency workers for a long time [8]. Polyethylene is relatively low loss up to mid-infrared frequency (18 THz) [9]. It has an intense sharp absorption band at the center of the submillimeter-wave band (2.1 THz) and the use of an alternative material as the window or lens is desirable in the region 1.65–2.65 THz. Polypropylene was first used in the submillimeter-wave region [9], [10] as a substitute for polyethylene. It is more easy to fabricate and its mechanical properties are more attractive. For dielectric applications in the 1-GHz region, it has been used in a rather parallel fashion as a substitute for polyethylene [8]. The loss angle, tan δ, at 1 GHz of both polyethylene and polypropylene is of the order of 10^{-4}.

In the strict sense, there is no pure polymer and they all show wide ranges of molecular weight. These semicrystalline materials are made of crystalline regions spread throughout an amorphous matrix and are quite inhomogeneous. The optical properties of all polymers (particularly the absorption coefficient) can vary markedly with the composition, history, and the temperature of the specimen [11]. Absorptions by polymers in the millimeter- and submillimeter-wave region arise from low-frequency intramolecular modes, intermolecular or lattice modes, amorphous region processes, relaxation or nonresonant processes, and from impurities accidentally or deliberately present in the material [7], [8]. The mid- and near-infrared absorptions arise from vibrations of highly localized groups of atoms [7]. For a long chain molecule chain-twisting motions give rise to absorptions in the far-infrared (submillimeter-wave region).

This paper presents new continuous data on the absorption coefficient, the real part of the dielectric permittivity, and the loss tangent for commercially available high-density polyethylene, polypropylene, polytetrafluoroethylene (Teflon), and poly-4-methylpentene-1 (TPX) over the frequency range 60–300 GHz.

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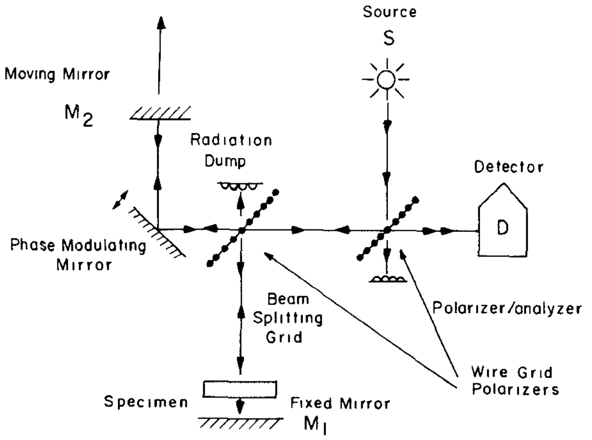

Fig. 1. The layout of a two-beam polarization interferometer for dispersive Fourier transform spectroscopy of solids. The radiation source is a high-pressure mercury vapor lamp. The polarizer/analyzer grid polarizes the incident beam and analyzes beams before reaching the detector. The beam-splitter grid splits the polarized beam and recombines them.

II. MEASUREMENT METHODS

In the millimeter-wave region, a quasi-optical technique, namely dispersive Fourier transform spectroscopy developed and perfected by the author [11]–[17], is used for measurements. Detailed descriptions of the dispersive Fourier transform technique, together with other techniques such as the Fabry–Perot open resonator technique, are given in [17] and [24]. Here, in this paper, a brief description of the dispersive FTS is outlined. The usual procedure in an optical technique is to measure directly the complex refractive index $\hat{n} = n - ik = n - i(\alpha/4\pi\tilde{\nu})$, where n is the refractive index, k is the absorption index, α is the familiar absorption coefficient in Np/cm, and $\tilde{\nu}$ is the wavenumber per cm. The wavenumber $\tilde{\nu}$ is related to the frequency ν via the speed of light c so that $\tilde{\nu} = C/\nu$. It is convenient to note that 1 wavenumber (cm^{-1}) = 30 GHz. The real and imaginary parts of the dielectric parameters ϵ', ϵ'', and $\tan\delta$ are calculated from

$$\begin{aligned}\epsilon' &= n^2 - k^2 = n^2 - (\alpha/4\pi\tilde{\nu})^2 \\ &= n^2 - (c\alpha/4\pi\nu)^2 \qquad (1)\end{aligned}$$

$$\epsilon'' = 2nk = (\alpha n)/2\pi\tilde{\nu} = (\alpha c n)/2\pi\nu \qquad (2)$$

$$\tan\delta = \epsilon''/\epsilon' \qquad (3)$$

with directly measured values of α and n.

The great advance in millimeter-wave spectroscopy came with the development of dispersive Fourier transform spectroscopy applied to a polarization two-beam interferometer. This was recently carried out very successfully by the author at the MIT National Magnet Laboratory [17]. The basis of the dispersive Fourier transform spectroscopy (DFTS) is to introduce the specimen into one of the active arms of the two beam interferometer. The usual procedure is to record a set of interferograms, one without the specimen and one with the specimen. The interferogram with the specimen incorporates a number of interference signatures. The basic interferometer layout is shown in Fig. 1. The source is a broad-band high-pressure mercury vapor lamp. The most suitable, sensitive and fast detector for broad-band millimeter-wave spectroscopy is the Rollin-type InSb hot-electron bolometer [18]. The wire-grid polarizers polarize the incident radiation from the lamp, then divides. The perpendicular component goes through and the parallel component reflects into the specimen arm. Beams reflect back from the fixed mirror in the specimen arm and from the scanning mirror and recombine at one of the wire grids (beam-splitter grid). Finally, both beams get analyzed by the polarizer/analyzer grid before reaching the detector. This beam-splitting technique allows a broader passband compared to a commonly used mylar beam splitter in a spectrometer and is particularly useful at millimeter wavelengths.

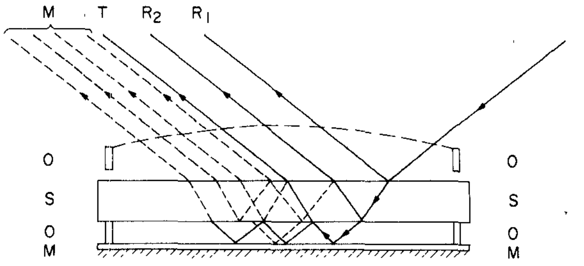

Fig. 2. The enlarged diagram of the specimen-holder fixed-mirror arm of the interometer. O, S, M represent dry air, solid sample, and mirror, respectively. A thin mylar film is shown above. It isolates the evacuated interferometer from the specimen holder. R_1 is the front specimen surface reflected beam, R_2 is the back specimen surface reflected beam, T is the transmitted beam, and M are multiply reflected beams. Beams are at normal incidence, but shown at inclined incidence for figure clarity.

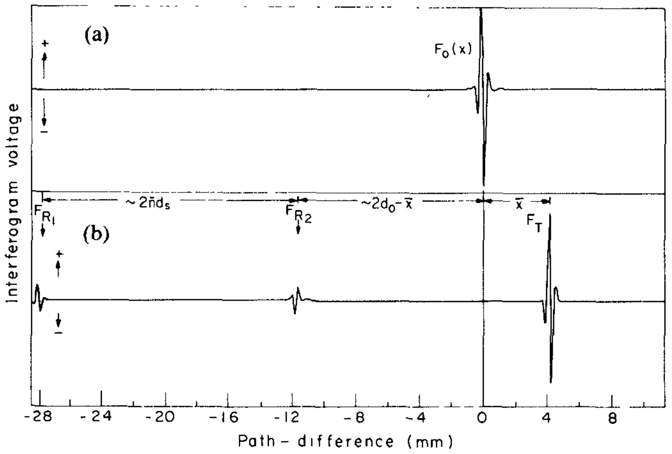

Fig. 3. Phase-modulated interferograms recorded using the polarization interferometer and a Rollin detector (a) reference interferogram $F_0(x)$ (b) with a TPX specimen of thickness = 5.761 mm. The interferogram with the specimen $F_S(x)$ signatures is a sum of $F_{R_1}(x) + F_{R_2}(x) + F_T(x) + F_M(x)$. The $F_M(x)$ signatures are not shown in the figure.

The enlarged version of the specimen holder can be seen in the Fig. 2. The plane-parallel slab of solid specimen rests on a support above the mirror in one arm of the interferometer. The specimen surfaces are also parallel to the mirror giving three plane-parallel interfaces: dry air (OS), specimen and dry air (SO), and dry air and mirror (OM). Phase modulated interferograms obtained without and with the specimen for a TPX sample can be seen in Fig. 3.

The reference interferogram $F_0(x)$ is recorded without the specimen. The specimen interferogram $F_S(x)$ with the specimen thickness d_s is a sum of interferogram signatures

$$F_S(x) = F_{R_1}(x) + F_{R_2}(x) + F_T(x) + F_M(x). \tag{4}$$

The $F_M(x)$ is the sum of all multiply reflected signatures (not shown in Fig. 3). For millimeter waves where one does not require the specimen thickness to be too small, signatures are well separated and signatures can be easily separated. In theory, any pair of signatures should provide an evaluation of the complex refractive index but it is preferable to use a strong reference signature $F_0(x)$ together with a strong $F_T(x)$ signature for full evaluations of the absorption coefficient and refractive index.

The complex transmission insertion loss $[\hat{\mathscr{S}}_T(\tilde{\nu})]^2$ is given by

$$\begin{aligned}[\hat{\mathscr{S}}_T(\tilde{\nu})]^2 &= \frac{\mathscr{F}[F_T(x)]}{\mathscr{F}[F_0(x)]} \\ &= [\hat{S}'(\tilde{\nu})]^2 \exp\left(-4\pi i \nu[\hat{n}(\hat{\nu})-1]d_s\right) \\ &= \hat{S}_T(\tilde{\nu})/\hat{S}_0(\tilde{\nu}) \end{aligned} \tag{5}$$

where $[\hat{S}'(\tilde{\nu})]^2$ represents the total surface reflection loss contributions, and $\hat{S}_T(\tilde{\nu})$ and $\hat{S}_0(\tilde{\nu})$ represent, respectively, Fourier's transformed complex spectra of $F_T(x)$ and $F_0(x)$ interferogram signatures. The refractive index and the absorption coefficient spectra are then calculated via an iterative procedure [12]. The specially constructed highly reproducible and stable, computer automated interferometer utilizes the full 60-mm radiation throughput in order to avoid diffraction effects. The final data are highly reproducible reflecting *five-figure accuracy* in the real part of the dielectric permittivity and the refractive index data and to about 1 percent accuracy in the absorption coefficient and loss tangent data.

The specimen sizes used in these measurements range from 60 to 75 mm in diameter and 50 to 75 mm in thickness. As the beam passes through the specimen twice in one of the active arm of the FTS two-beam interferometer, this is equivalent to a specimen of twice the thicknesses used.

These specimens were prepared by employing a diamond fly cut in a vacuum chuck machine. The parrallelism of surfaces was close to a few seconds of an arc. The flatness of surfaces was also close to a flatness required for optical frequencies. The above procedures of the sample preparation is essential for a relatively soft, solid material like polymer to avoid contamination.

III. Results and Discussions

In polymers, small molecules make up a typical molecular crystal which are bound together by strong valence forces, but they are held in their correct places in the lattice only by the much weaker Van der Waals forces. The vibration of the ensemble can be analyzed by the classid methods described in the book by Born and Huang [19]. Millimeter- and submillimeter-wave spectroscopic techniques are capable of revealing intimate details of the conformation of chains and the interatomic forces operative in and between them. The motions of pendant side chains lead to vibrational bands well into the far-infrared in spectral regions where they are intermingled with lattice-type vibrational bands, and also with the complex broad-band absorption characteristic of the amorphous solids and liquids. The submillimeter-wave and far-infrared absorptions of polymers show two distinct types of absorption; the sharp lattice-type vibration band absorption arising in the crystalline region and the complex broad-band absorption due to the amorphous continuum absorption originating in the millimeter-wave range. This is analogous to the broad-band absorptions characteristic of polar and nonpolar liquids. Additionally, the vibrational spectrum of polytetrafluorethylene have shown a band around 420 GHz [21]. One of the reasons why millimeter-wave spectroscopy of polymers has been neglected is because solid-state physicists are more used to measurements of phonon absorptions for single crystals and are not used to handling polymers. One can define phonons, each with a characteristic frequency, ν and wave-vector K, when ν is plotted against K, and it gives a dispersion diagram characterized by a series of branches, which can be divided into two classes, the optical and acoustic branches.

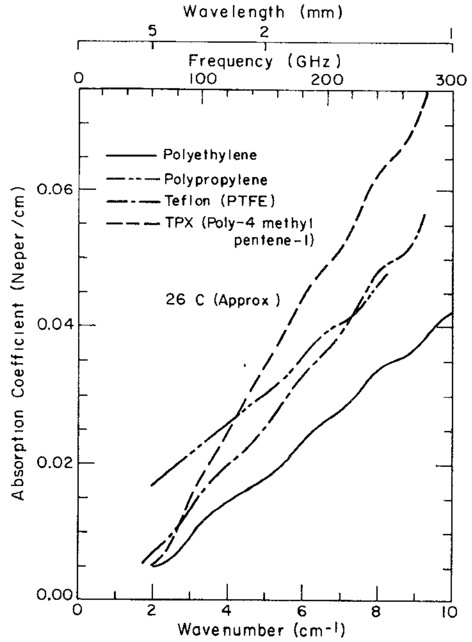

Fig. 4. Comparison of room-temperature absorption coefficient spectra for commercially available specimens of polyethylene, polypropylene, polytetrafluorethylene (Teflon), and poly-4-methylpentene-1 (TPX) over the frequency range 60–300 GHz. The absorption is very low for all four, but shows a tendency of increase with increasing frequency. Polyethylene exhibits the lowest loss characteristics. It is probably *the* lowest absorption millimeter-wave material.

The new millimeter-wave continuous absorption coefficient data for high-density polyethylene, polypropylene ("isotactic") polytetrafluorethylene, and poly-4 methylpentene-1 over the frequency range 60–300 GHz are shown in Fig. 4. All four materials exhibit very low absorption values, but the trend of increasing values of the absorption reflects the presence of the amorphous continuum. The polyethylene can now be classified as *the* lowest loss material in the entire millimeter-wavelength range. Absorption values are as low as 0.003 Np/cm at 60 GHz and increases to 0.042 Np/cm at 300 GHz. It is followed by Teflon with a value of about 0.006 Np/cm at 60 GHz. This trend does agree closely with microwave measurements made at 35 GHz by the author [22]. Our polypropylene specimen is a commercially available "isotactic" material. Usually, commercially available polypropylene contains far more impurities than does polyethylene. There are not only such things as anti-oxidants and plasticizers, which are normally added to improve the mechanical and electrical properties of the material, but also inadvertent impurities, such as catalyst residues. Some of these impurities are ionic, which give rise to heavy "reststrahlen"-type absorption above 3000 GHz, and for this reason many commercially available polypropylene samples are quite opaque in the submillimeter and far-infrared region. The trailing edge of this absorption may also produce loss in the millimeter-wave band. The absorption of polypropylene in the 60–300 GHz range is higher than the polyethylene by an amount of about 0.012 Np/cm, as if the polyethylene absorption spectrum is raised by 0.012 Np/cm. A comparison with the 35-GHz value [22] reveals a different situation. At 35-GHz, teflon exhibits higher loss than the polypropylene but our polypropylene shows more loss at 60 GHz compared to Teflon.

The TPX is a relatively new polyolefine based on a poly-4-methyl pentene-1 and known under the trade name TPX by its manufacturer Imperial Chemical Industries, UK. This material has some remarkable properties that made it particularly useful in the construction of submillimeter-wave equipment. This material can be moulded under pressure at elevated temperatures to give a solid product, very transparent in the visible region and which is hard and resistant to deformation by heat. From our results, as shown in Fig. 4, the absorption coefficient at 60 GHz has almost the same value as polyethylene, but the difference rapidly rises with increasing frequency. Beyond 150 GHz, it exhibits higher loss compared to the other three. TPX is a better candidate as a window and lens material in the submillimeter and far-infrared wavelength region because it does not have the sharp, strong absorption characteristics of the polyethylene at 2190 GHz.

The continuous millimeter-wave data for the real part of the dielectric permittivity for polyethylene and polypropylene are shown in Fig. 5. The general trend is the fall or decrease of ϵ' values with increasing frequency which is consistent with the increase of absorption with increasing frequency. These are high precis on data. A comparison of these results with a 35-GHz [22] value (2.316 ± 0.004) shows an excellent agreement for polyethylene. For polypropylene, there is a slight disagreement. The 35-GHz value [22] is 2.254 ± 0.004. This is consistent with our argument on commercially available polypropylene, mentioned above.

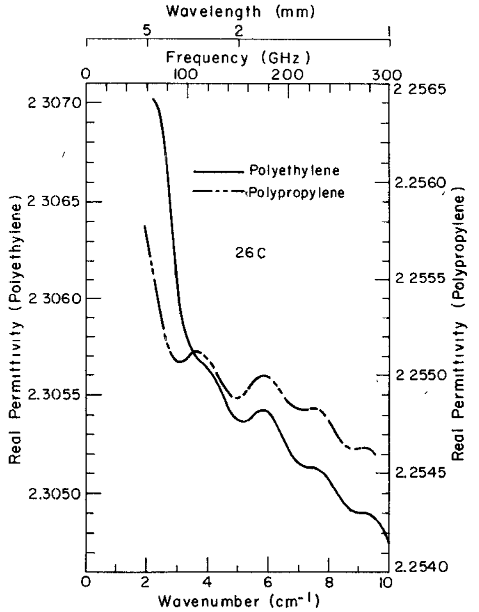

Fig. 5. Spectra for the real part of dielectric permittivity for polyethylene and polypropylene over the 60–300 GHz range at 26°C. Refractive index values are essentially the square root of the real part of the dielectric permittivity values.

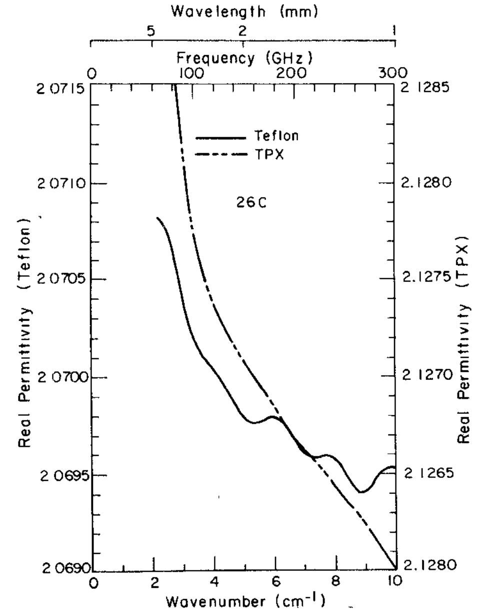

Fig. 6. Spectra for the real part of the dielectric permittivity for polytetrafluoroethylene (Teflon) and poly-4-methylpentene-1 (TPX) at 26°C over the frequency range 60–300 GHz.

The continuous data for the real part of the dielectric permittivity for polytetrafluorethylene (Teflon) and poly-4-methylpentene-1 (TPX) are shown in Fig. 6. A similar trend of decrease of the real part of the permittivity with increasing frequency is observed. The slope of the TPX curve is slightly larger compared to the Teflon curve. Excellent agreement with the author's past 35-GHz [22] data can be noticed (2.126 ± 0.004) for TPX. The only other data available for Teflon was at 50 GHz from the work of Culshaw and Anderson [23]. This early data (2.052 ± 0.02) has a much higher error but agrees very well with our present millimeter-wave data.

In polytetrafluorethylene, bulky fluorine atoms are sufficiently active to prevent the linear chain or folded linear

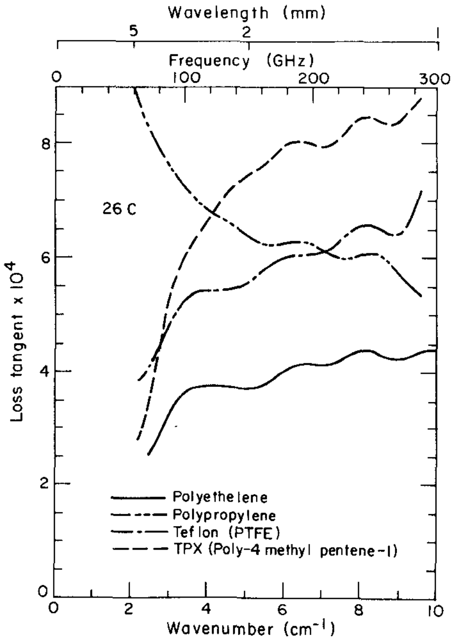

Fig. 7. Comparison of room-temperature loss tangent spectra of polyethylene, polypropylene, polytetrafluorethylene (Teflon), and poly-4-methylpentene-1 (TPX) over the frequency range 60–300 GHz. Again, polyetheline shows lowest loss tangent values over the entire millimeter wavelength range.

chain conformations from being the lowest energy configurations and the chain adopts a helical disposition. As this nonpolar molecule is integrated over a significant length of coil, the overall dipole moment vanishes, and this polymer can be expected to become a very low-loss material. The loss is extremely low for unsintered Teflon [24]. The new loss tangent data for all these four nonpolar polymers over the frequency range 60–300 GHz are shown in Fig. 7. The variation of the loss tangent values are typically 5×10^{-4} rad. Again, polyethylene exhibits the lowest loss followed by Teflon. The 35-GHz loss-tangent data of the author [22] for polyethylene is 134 ± 7 microradians. If we read the value of loss tangent at 70 GHz for polyethylene from the new continuous data (Figure 7), we get a value of 250 microradians. By observing the trend of the curve, it agrees quite well. The polypropylene again shows a big discrepancy with the author's earlier measurement at 35 GHz [22]. The earlier 35 GHz value for polypropylene is 154 ± 8 microradians. The 50-GHz loss tangent of Culshaw and Anderson for Teflon is 315 ± 26 microradians which is in good agreement with our continuous data. Our value of the loss tangent for Teflon at 70 GHz is 380 microradians. The loss tangent curve for poly-4-methylpentene-1 (TPX) shows a lower value compared to Teflon below 80 GHz. At 60 GHz, it is as low as 280 microradians. A comparison of this data with the author's early open-resonator measurement [22] at 35 GHz on TPX shows a slight discrepancy. The 35-GHz open-resonator value for the loss tangent was 427 ± 21 microradians. We do not, at present, have any explanation for this discrepancy.

The refractive data are not shown here. But for a low-loss material, it is essentially the square root of the real part of the dielectric permittivity values.

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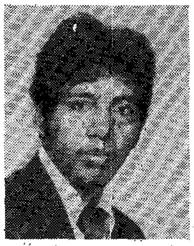

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