FHWA NJ 2000-04

# Recycled Plastic Fibers for Asphalt Mixtures

FINAL REPORT
August 1999

Submitted by

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In cooperation with

New Jersey
Department of Transportation
Division of Research and Technology
and
U.S. Department of Transportation
Federal Highway Administration

# Disclaimer Statement

TECHNICAL REPORT STANDARD TITLE PAGE

1. Report No.
FHWA 2000-04

2. Government Accession No.

3. Recipient's Catalog No.

4. Title and Subtitle
Recycled Plastic Fibers for Asphalt Mixtures

5. Report Date
August 1999

6. Performing Organization Code
NJIT & CAIT/Rutgers

7. Author(s) Dr. Mohammed Labib and Dr. Ali Maher

8. Performing Organization Report No.
FHWA 2000-04

9. Performing Organization Name and Address
New Jersey Department of Transportation
CN 600
Trenton, NJ 08625

10. Work Unit No.

11. Contract or Grant No.

12. Sponsoring Agency Name and Address
Federal Highway Administration
U.S. Department of Transportation
Washington. D.C.

13. Type of Report and Period Covered
Final Report
3/6/1996 - 3/31/1999

14. Sponsoring Agency Code

15. Supplementary Notes

16. Abstract

The use of recycled fibers in asphalt mixtures were found to be beneficial in some cases only such as when fishing nets were used. In these successful cases, the fibers could be uniformly and consistently incorporated into the asphalt mixture without segregation or introduction of excessive air voids. Fibers typically obtained from recycling operations such as from carpets and car seats were difficult to use with the dry mixing process that was developed at NJIT. The latter sources of fibers produced waste that was entangled. This is why the results were inconsistent with regard to the effect of fibers on the performance of the mixture. Available recycled fibers did not produce consistent results as judged by the Marshall test. The final conclusion of the program was to pursue a systematic study primarily based on using the Superpave design Schedule. The final approach was to use virgin fibers first to clearly understand how fibers affect the mechanical properties of the mixture, and then apply the results to the use of recycled fibers – all the work was to follow the Superpave design approach. A comprehensive review of the results is included in this report.

17. Key Words
Recycled Fibers, Asphalt Mixtures, Fibers, Fishing Net, Marshall Stability, and Recycling

18. Distribution Statement

19. Security Classif (of this report)
Unclassified

20. Security Classif. (of this page)
Unclassified

21. No of Pages
93

22. Price

Form DOT F 1700.7 (8-69)

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# ACKNOWLEDGMENTS

This project was conducted in cooperation and under sponsorship of the New Jersey Department of Transportation (NJDOT). The principal investigators express their gratitude to the NJDOT for funding the research described herein.

# Recycled Plastic Fibers for Asphalt Mixtures

## Dr. M. E. Labib

## I. Summary and Conclusions:

The objective of the program was to explore the applicability of using recycled plastic fibers to improve the mechanical properties and the performance of asphalt mixtures. The program was limited in scope, with the initial intention to explore the potential of this application. The total funding at $24,674. The project was only allocated for one year. Mr. Song Gu, a graduate student, was assigned to pursue this program. Mr. Gu has left NJIT to pursue a professional engineering career in the area of structural analysis with a consulting firm in New Jersey.

***The main results of the program were as follows:***

1. A complete literature survey was conducted to estimate the availability of fibers for this major application. Production information of non-cellulose fibers in the United States was compiled and reviewed - Attached as Results Set I.
2. An elaborate review of the literature regarding the state of the art of fiber-matrix interface and its implication of the micro- and macro-mechanics of composite materials was conducted and summarized in the form of a report. Attached as Results - Set II
3. A dry process for mixing recycled fishing net fibers for making asphalt mixture for initial investigation was developed during this program - Attached as Results Set III.
4. Preliminary experimental evaluations of asphalt mixtures made with various recycled fibers using the Marshall stability test were conducted. This part is described in this final report summary. The results were variable and inconsistent.
5. An elaborate test program for evaluating fiber-containing asphalt mixtures using Superpave schedule was developed for future work. Attached as Results Set IV.

**Synopsis:** The use of recycled fibers in asphalt mixtures were found to be beneficial in some cases only such as when fishing nets were used. In these successful cases, the fibers could be uniformly and consistently incorporated into the asphalt mixture without segregation or introduction of excessive air voids. Fibers typical obtained form recycling operations such as from carpets and car seats were difficult to use with the dry mixing process that was developed at NJIT. The latter sources of fibers produced waste that was entangled. This why the results inconsistent with regard to the effect of fibers on the performance of the mixture. Available recycled fibers did not produce consistent results as judged by the Marshall test. The final conclusion of the program was to pursue a systematic study primarily based on using the Superpave design Schedule. The final approach was to use virgin fibers first to clearly understand how fibers affect the mechanical properties of the mixture, and then apply the results to the use of recycled fibers - all the work was to follow the Superpave design approach. A comprehensive review of the results is included in this report.

## II. Results and Discussion

The results of this program will be divided into several sections as follows:

### 1. Information regarding US organic fiber production:

A market research regarding the type and nature of fibers produced in the US was conducted and summarized. It is estimated that about 3,000 million tones of plastic fibers is produced in the US every year. These are as follows: 68% nylon, 19% polypropylene, 10% polyester and 3% other types. Based on this information, we decided to explore both nylon and polypropylene. The results of this research is attached as Results Set I.

### 2. Literature research regarding the characterization of fiber surfaces in polymer matrix composites and implication such properties on the mechanical and performance properties of such composites:

Since the asphalt-fiber interface case is similar, in principle, to the fiber-polymer case, we devoted part of the investigation to defining characterization methods that can be used in the project. This situation of fibers in asphalt mixture is, however, much more complex compared to the polymer composites case. The detail of this research is attached as Results Set II.

### 3. The development of durable asphalt pavements using fishing nets as reinforcement – mixing process development and initial results:

The study of evaluating the utility recycled fibers in asphalt mixtures started with the use of fishing net fibers, and was expanded during this program. We have studied the effect of fiber type on the mechanical and physical properties as judged by the Marshall stability results and by the voids analysis of asphalt mixtures. Based on the results, polypropylene fibers produced better results compared to nylon fibers. The details of this analysis are attached as Results Set III.

### 4. Summary of preliminary experiments:

Samples of nylon recycled plastic fibers were obtained from a carpet recycling company in Atlanta. The fibers were incorporated at different percentage into asphalt mixture using the methods developed for fishing net fibers, as described in Results Set III. Marshall testing was performed on the mixture produced. The results exhibited wide variability and in most cases deterioration of the stability values compared to control samples made without fibers. The reason for this result, in comparison with the fishing net fiber case, was attributed to the inability of incorporating the fibers into the mixture in a uniform manner and to the nature of this type of recycled fibers. The carpet recycled fiber received in this case were all entangled and lumpy and could not be readily used for this application. Based on the above results we decided to follow a methodical approach where virgin fibers with known properties to be used and the mixture design to followed according to the Superpave methodology.

5. Test program for hot melt asphalt mixtures using the Superpave schedule:

A detailed test program and strategy were formulated to continue this program in order to properly determine the benefits of using fibers in mixtures. This strategy is attached as Results Set IV and contains an introduction, importance of using Superpave schedule Level 3, study scheme and experimental work needed to complete a well-thought project.

In conclusion, the project has fulfilled its objectives with respect to examination of the feasibility of using recycled fibers in asphalt pavement. Presently, it appears that available recycled fibers are not suitable for application in asphalt mixture. The properties of such recycled fibers are variable; therefore, a standard and validated process to use in asphalt mixture is expected to be cumbersome or even impossible. An expanded study is proposed to evaluate the utility of fibers in asphalt pavement, especially with the application of the Superpave design methodology. Once the requirement for using fibers in mixture are determined, a set of criteria would be developed for using recycled fibers. It is expected that recommendation to recycling operation would be made to prepare the fibers with specification to renders them suitable for use in asphalt pavement. We do not foresee the use of recoiled fibers in asphalt pavement at the present state of the art.

# RESULTS SET I

# PRODUCTION INFORMATION ABOUT U.S. ORGANIC FIBERS-NONCELLULOSIC

Department of Civil Engineering, NJIT

October 20, 1996

Establishments primarily engaged in manufacturing noncellulosic or synthetic fibers comprise the manmade organic fibers industry. The fibers are created in the form of monofilament, yarn, staple, or tow suitabe for further manufacturing on spindles, looms, knitting machines, or other textile processing equipment. Sythetic fibers accounted for about 90% of manmade fibers output in 1993. Manmade fibers constituted approximatly 25% of the larger U.S. synthetic materials industry, which also encompassed plastics and rubbers.

Synthetic materials, in turn, represented about 25% of the overall $300 billion per year U.S. chemical industry.

The portions of each part of application of sythetic noncellulosics fibers are shown in Fig 1.

The major synthetic noncellulosics are polyster, nylon, acrylic, and the olefins. The volumes of production and capacity of these items can be seen in Table 1. and Table 2. Each item percentage of the total synthetic noncellulosics materials is shown in Fig 2. And Fig 3 and 4 are ploted to show the percentages of each uses from polyster and nylon separately.

Over the past decade, olfins fibers (polypropylene and polyethylene) have been the fastest-growing manufactured fiber category. Growth has been spurred by the increasing use of olefins in a variety of products; more than 50% of demand is for carpets and carpet backing, primarily for commercial uses. In addition to various industrial applications, other uses of olefins are for nonwoven products such as disposable fabrics and medical garments.

Acrylic is used in apparel (sweaters, fleecewear, and hosiery). Acrylic fibers compete with polyster and cotton in apparel uses. Home furnishings (primarily blankets and draps) and industrial uses (filters, and reinforcements for friction products such as brake linings) account for the remaining acrylic demand.

Table 3 refers to the production of U.S. carpet fiber market in 1993

According to Modern Plastics Jan. 1995, in the recycled PET markets in 1993 and 1994, the volumes of recycled fibers are 210 and 240 million pounds respectively.

The largest company competing in the synthetic fibers industry in the early 1990s, E.I. du Pont de Nemours of Delaware planed to develop nylon recycling technology to begin marketing recyclable fibers by 1997, the company hoped to eventually corner 85% of the used nylon market.

The second manufacturer of synthetic fibers in the early 1990s, Hoechest Celanses Corp., introduced a 100% recyclable, all-polyster carpet system in 1993.

## References

1. Standard & Poor's Industry Surveys. NY S&P's Corporation 1996
2. Encyclopedia of American Industries. Vol.1, Gale Research Inc. 1994
3. Modern Plastics Jan. 1995
4. Carpet & Rug Industry Oct. 1993

Table 1

**U.S. manufactured fiber production**
(In millions of pounds)

| | Rayon & Acetate | | Noncellulosic fibers | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | Yarn & monofilaments | | | | Staple & tow | | | | |
| Year | Filament yarn | Staple & tow | Nylon | †Olefin | Polyester | Total | Nylon | Acrylic | Polyester | Total | Grand total |
| 1993 | 227 | 278 | 1,700 | 1,659 | 1,284 | 4,643 | 959 | 433 | 2,274 | 4,148 | 8,791 |
| 1992 | 220 | 275 | 1,652 | 1,528 | 1,269 | 4,449 | 904 | 439 | 2,307 | 4,124 | 8,573 |
| 1991 | 213 | 273 | 1,666 | 1,408 | 1,208 | 4,282 | 869 | 454 | 2,203 | 3,984 | 8,753 |
| 1990 | 206 | 299 | 1,672 | 1,417 | 1,105 | 4,194 | 990 | 506 | 2,090 | 3,991 | 8,690 |
| 1989 | 218 | 363 | 1,759 | 1,257 | 1,209 | 4,225 | 981 | 543 | 2,385 | 4,290 | 9,096 |
| 1988 | 214 | 400 | 1,728 | 1,204 | 1,229 | 4,160 | 942 | 588 | 2,452 | 4,346 | 9,140 |
| 1987 | 191 | 414 | 1,697 | 1,134 | 1,179 | 4,010 | 992 | 592 | 2,362 | 4,306 | 8,921 |
| 1986 | 215 | 404 | 1,606 | 1,058 | 1,173 | 3,836 | 908 | 616 | 2,132 | 3,992 | 8,447 |
| 1985 | 205 | 353 | 1,515 | 955 | 1,321 | 3,790 | 828 | 631 | 2,020 | 3,773 | 8,121 |
| 1984 | 239 | 390 | 1,577 | 747 | 1,200 | 3,524 | 835 | 671 | 2,192 | 3,947 | 8,100 |

*Includes other. †Polyethylene and polypropylene.
Source: *Fiber Organon.*

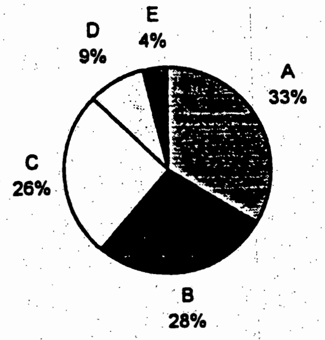
Table 2

**U.S. manufactured fiber capacity**
(In millions of pounds)

| | Rayon & acetate | | Noncellulosic fibers | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | Yarn & monofilaments | | | | Staple & tow | | | | | |
| Year | Filament yarn | Staple & tow | Nylon | *Olefin | Polyester | Total | †Nylon | Acrylic | Polyester | Olefin | Total | Grand total |
| E 1995 | 233 | 440 | 2,128 | 1,849 | 2,304 | 6,281 | 1,089 | 475 | 2,822 | 693 | 5,079 | 12,033 |
| E 1994 | 233 | 375 | 2,119 | 1,795 | 2,268 | 6,182 | 1,031 | 475 | 2,722 | 662 | 4,890 | 11,680 |
| 1993 | 234 | 375 | 2,052 | 1,656 | 2,034 | 5,742 | 1,073 | 482 | 2,763 | 577 | 4,895 | 11,246 |
| 1992 | 224 | 360 | 1,980 | 1,889 | 1,601 | 5,470 | 1,101 | 494 | 2,877 | 567 | 5,039 | 11,093 |
| 1991 | 220 | 325 | 2,002 | 1,800 | 1,374 | 5,176 | 1,141 | 487 | 2,603 | 555 | 4,786 | 10,507 |
| 1990 | 219 | 322 | 2,024 | 1,761 | 1,341 | 5,126 | 1,144 | 446 | 2,578 | 520 | 4,688 | 10,355 |
| 1989 | 222 | 432 | 2,018 | 1,580 | 1,320 | 4,918 | 1,150 | 641 | 2,464 | 500 | 4,755 | 10,397 |
| 1988 | 244 | 469 | 1,897 | 1,470 | 1,376 | 4,743 | 1,137 | 642 | 2,584 | 486 | 4,849 | 10,305 |
| 1987 | 244 | 465 | 1,827 | 1,402 | 1,328 | 4,557 | 1,137 | 652 | 2,522 | 482 | 4,793 | 10,059 |
| 1986 | 244 | 465 | 1,797 | 1,326 | 1,422 | 4,545 | 1,077 | 637 | 2,491 | 457 | 4,662 | 9,916 |
| 1985 | 269 | 515 | 1,868 | 1,329 | 1,471 | 4,668 | 1,067 | 631 | 2,785 | 407 | 4,890 | 10,342 |

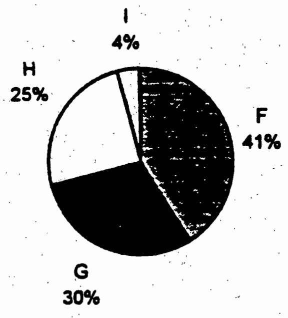
E–Estimated. *Polyethylene and polypropylene. †Includes aramid.
Source: *Fiber Organon.*

**Fig.1 Fibers Products Distribution**



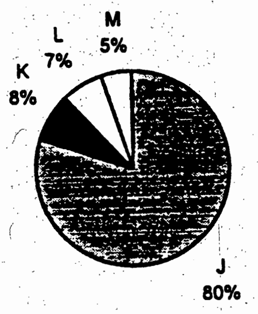
A- Floor covering, 3216 mil lbs
B- Industrial production & other consumer goods (tires, rope, medical and surgical supplies, electrical equipment, reinforced plastics and coated and protected fibers), 2649 mil lbs
C- Apparel, 2459 mil lbs
D- Home textile, 851.3 mil lbs
E- Exports, 378.4 mil lbs

**Fig.2 Distribution of different kinds of sythetic fibers**



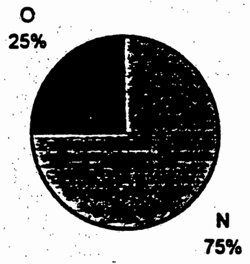
F- Polyster
G- Nylon
H- Olefins
I- Acrylic

**Fig.3 Distribution of the products of polyster fibers**



J- Textiles, apparel, home furnishings
K- Rubber reinforcement
L- Other industrial products
M- Carpets

**Fig.4 Distribution of the products of nylon**



N- Carpeting
O- Apprel. noncarpet, home furnishings and industrial uses (mainly tires, other rubber products and belts and rope)

Table 3 Production of US Carpet Fiber in 1993 year

| Type | Volume (mil lbs) | Percent of the total carpet fibers (%) |
|---|---|---|
| Nylon | 1900 | 68 |
| Polypropylene | 527 | 19 |
| Polyster fiber | 280 | 10 |
| Others including wool and cotten | 66 | 3 |
| Total | 2773 | 100 |

Table 1

Table 2

A- Floor covering, 3216 mil lbs
B- Industrial production & other consumer goods (tires, rope, medical and surgical supplies, electrical equipment, reinforced plastics and coated and protected fibers), 2649 mil lbs
C- Apparel, 2459 mil lbs
D- Home textile, 851.3 mil lbs
E- Exports, 378.4 mil lbs

F- Polyster
G- Nylon
H- Olefins
I- Acrylic

J- Textiles, apparel, home furnishings
K- Rubber reinforcement
L- Other industrial products
M- Carpets

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# RESULTS SET II

# PRESENTATION

# LITERATURE RESEARCH OF CHARACTERIZING FILLER OR FIBER SURFACES IN POLYMER COMPOSITES

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New Jersey Institute of Technology

May 1997

# LITERATURE RESEARCH OF CHARACTERIZING FILLER OR FIBER SURFACES IN POLYMER COMPOSITES

## 1. Introduction

1.1. Polymer composites with fillers (fibers)
1.2. Purpose of using surface-reactive chemicals or surface treatments
1.3. Objective of characterization of filler (fiber) surfaces in the composites
1.4. Analytical techniques

## 2. Application of spectroscopic techniques in the characterization

2.1. Introduction
2.2. Application of X-ray photospectroscopy in the characterization
2.3. Application of Secondary Ion Mass Spectroscopy in the characterization
2.4. Comparison of XPS with SIMS
2.5. Application of Fouriour Transform Infrared Spectroscopy

## 3. Transmission Electron Microscopy and its Applications

3.1. Introduction
3.2. Basic principles
3.3. TEM operation
3.4. Application and Examples

# 1. INTRODUCTION

## 1.1. Polymer composites with fillers (fibers)

Introducing fillers (fibers) into polymers may substantially modify the properties and cost of material.

Ex1: Carbon fibers constituting a material essentially characterized by a high specific strength and stiffness, in comparison with the other currently available engineering materials. These fibers are mainly used as reinforcing agent in composites along with a light-weight matrix, commonly an epoxy resin.

Ex2: Silica is dispersed in methyl methacrylate, which is commonly used for moulding into articles such as kitchen sinks, baths and bathroom fitments, to obtain strong chemical bonding between fillers and the matrix, and to achieve the improved mechanical properties of the final component.

### Most commonly used fillers :

Silica, aluminum hydroxide, calcium carbonate, alumina silicates, titanium dioxide.

### Most commonly used surface treatment of fibers:

Oxidative, amine.

## 1.2. Purpose of using surface-reactive chemicals or surface treatment

### Using surface chemicals or surface treatments (for fibers):

For optimization the interaction between the filler and matrix, including:

1. Enhancing the strength of adhesion between filler (fiber) and matrix;
2. Improving the wetting by the polymer, and hence the dispersion within the polymer .

### Surface-reactive chemicals having a single functionality:

Reactive towards the filler surface and to improve the wetting.

Ex: Steric acid used to surface modify carbonate for improving the rheology when filling into polypropylene

### Surface-reactive component having dual functionality:

Being capable of reaction with the filler surface, and also cross-linking into the polymer matrix to form continuous chemical bonding between the filler particles and the polymer matrix.

Ex: 3-(trimethoxysilyl) propyl methacrylate (MPS) used in methyl methacrylate filling with silica.

## 1.3. Objective of characterization of filler (fiber) surfaces in the composites

### To achieve the work below:

Design the surface modifier with functional groups
Determination of adhesion in surface
Comparison of surface and matrix compositions
Surface failure analysis

### We should have:

A comprehensive analysis of all the chemical moieties at the filler surface and their precise special arrangement.

### Acid-base theory:

Acid-base interactivity is normally used for the surface chemistry to predict melt and composite properties

### Adhesion mechanism for fiber surface treatment

1. Increasing the mechanical interlocking and physical bonds;
2. Improving and control the interfacial chemical bonding.

## 1.4. Analytical techniques

The techniques have been divided into two groups.

### First groups:

To characterize the surface of fillers by understanding their reactivity with probe molecules.

Including: flow microcalorimetry, inverse gas chromatography.
(which are not discussed here)

### Second groups:

Spectroscopic techniques, which uniquely provide elemental and chemical analysis of only the outer few nanometers of the surface under investigation.

### Including:

XPS, SIMS and DRIFT FTIR.

# 2. Application of spectroscopic techniques in the characterization

## 2.1. Introduction

### Technique requirement for surface characterization:

Surface modification of base filler particles can range from the absorption of less than one monolayer of a surfactant to the precipitation of a coating many nanometres (10E-9 M) thick.

Surface composition analysis therefore requires methods that posses sampling depths of order of 1-10 nm and which can characterize both inorganic and organic species.

### Ultrahigh vacuum (UHV)-based surface spectroscopic techniques close to meeting the requirements:

X-ray phontonelectron spectroscopy
Secondary ion mass spectrometry (static)

Because of charging problems with highly insulating powers and beam damage to organic materials, Auger electron spectroscopy (AES) fails in this field.

### Other spectroscopy techniques most commonly used:

Diffuse reflectance Fourier transform infrared spectroscopy

### Other physical techniques commonly used:

Transition electron microscopy
Scanning electron microscopy

## 2.2. Application of X-ray photospectroscopy in the characterization

### Basic theory:

In XPS, a beam of soft x ray (1-5ke V) illuminates the material to be studied and the energy spectrum of the resulting photoemitted electrons is measured by a suitable spectrometer. Each x-ray photon is usually completely absorbed by a single electron in the sample, providing enough of the escaping electron is :

$$Ee = hv - Eb$$

Where Ee is the kinetic energy of the escaping electron, hv is the x-ray photon energy and Eb is the binding energy of the electron in the material.

If the incident x-ray energy is known and the escaping electron energy is measured, the original binding energy can be calculated. Since each element has a unique set of binding energies, the binding energy in turn determines the element from which the electron came. These binding energies can be calculated or determined experimentally from x-ray absorption or emission measurements.

One of the most valuable features of XPS is its ability to distinguish different chemical bonding configurations as well as different elements.

## Special features of XPS:

1. The least destructive of all the electron or ion spectroscopy techniques.
2. Surface sensitivity (absolutely sensitivity between 0.01-0.3% at., depending on the element), but not suitable for trace analysis.
3. Quantitative and chemical state analysis capabilities.
4. Detect all elements except hydrogen and helium .
5. Relatively poor spatial resolution, compared to electron-impact and ion-impact techniques.
6. Depending on the element XPS can be a slow technique if the extent of chemical detail to extracted is large. Analysis times may vary from a few minutes to many hours.

## Application in surface characterization:

1. Examination for and identification of surface contaminants.
2. Evaluation of thin-film coatings.
3. Failure analysis for adhesion between components.
4. Effectiveness of surface treatments of polymers and plastics.

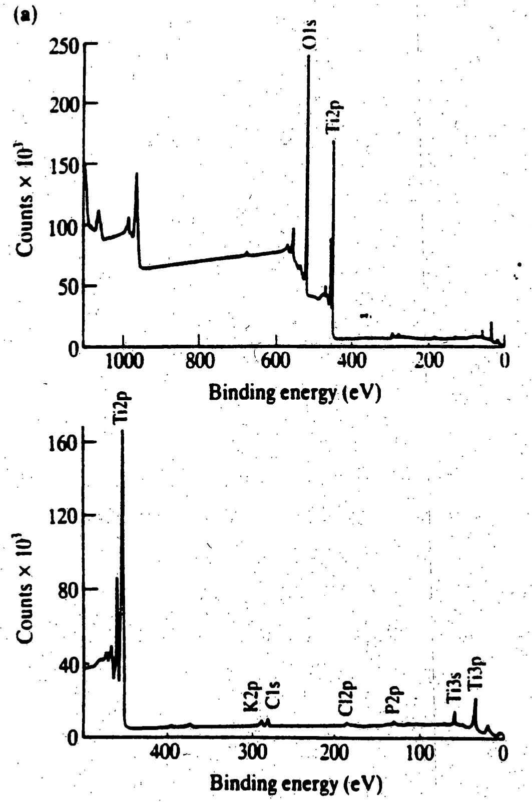

**Figure** (a) XPS survey spectra of uncoated commercial $TiO_2$ pigment.

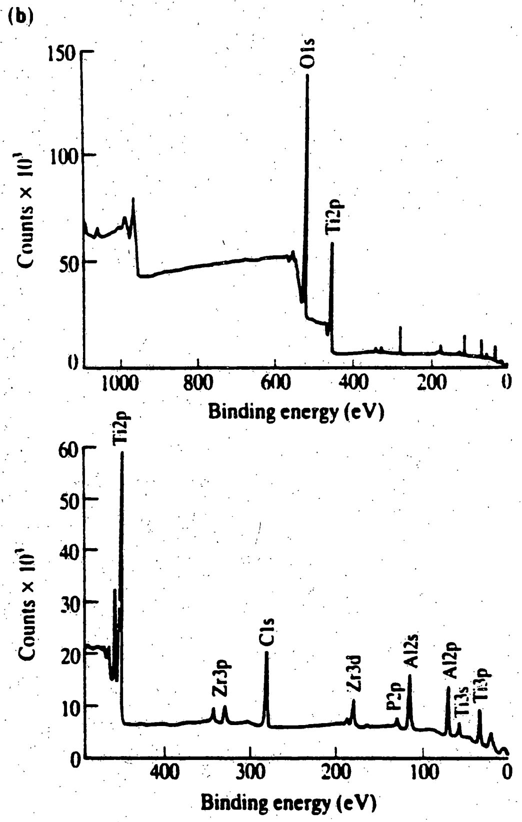

**Figure** (*continued*) (b) XPS survey spectra of coated commercial $TiO_2$ pigment. Note the marked attenuation of Ti 2p signal following coating.

## 2.3. Application of Secondary Ion Mass Spectroscopy in the characterization

### Basic principle

Static SIMS entails the bombardment of a sample surface with an energetic beam of particles, resulting in the emission of surfaces atoms and clusters. These ejected species subsequently become either positively or negatively charged and are referred to as secondly ions. The secondly ions are the actual analytical signal in SIMS. A mass spectrometer is used to separate the secondly ions with respect to their charge-to-mass ratios. The atomic ions give an elemental identification, whereas the clusters can information on the chemical groups.

The mass spectrum of the clusters obtained represents a fingerprint of the compounds analyzed. The data may be acquired over relatively small areas(um or less) for a localized analysis or larger areas (mm) for a macrocharacterization. Further, by monitoring a particular charge-to-mass ratio (i.e. a particular chemical group), one can obtain chemical maps depicting the lateral distribution of a specific fragment or compound.

### Special Features of SIMS (static)

1. High surface sensitivity.
2. Complete microchemical analysis by providing mass spectral data (both positive and negative spectrometry), as well as chemical mapping.

# Applications in surface characterization

1. Comparison of surface and bulk compositions.
2. Failure analysis.
3. Determination of adhesion or delamination mechanisms.

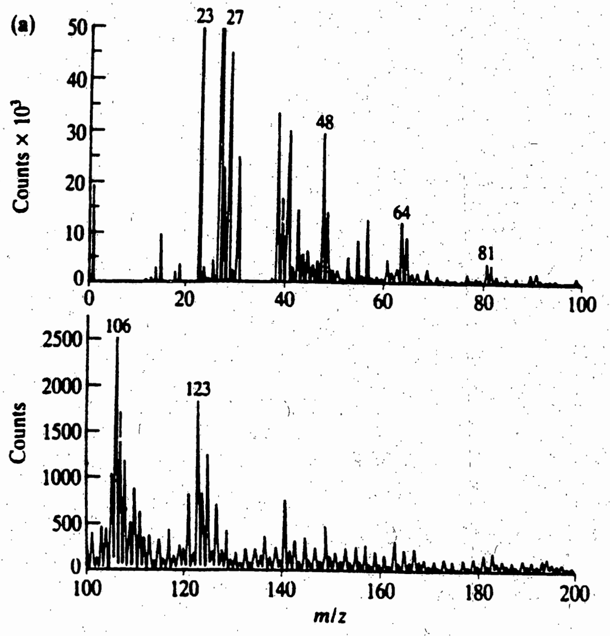

**Figure** ToF SIMS spectra from coated pigment giving XPS spectrum of Fig. 3.9~~(b)~~: (a) Positive ion spectrum; annotated peaks are due to $Na^+$ (*m/z* 23), $Al^+$ (27), $Ti^+$ (48), $TiO^+$ (64), $TiO(OH)^+$ (81), $ZrO^+$ (106) and $ZrO(OH)^+$ (123).

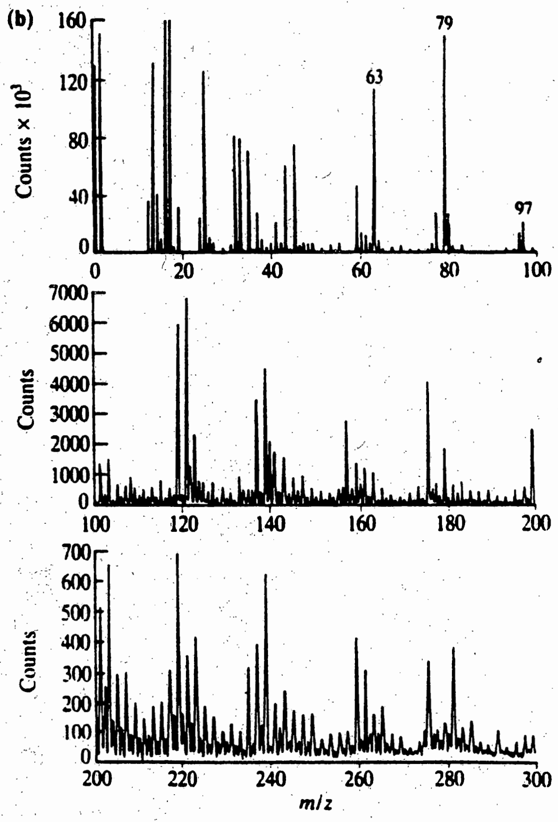

**Figure** (*continued*) (b) Negative ion spectrum: annotated peaks are due to $PO_2$ (63), $PO_3^-$ (79), $H_2PO_4^-$ (97); all other peaks are molecular fragments from organic species.

## 2.4. Comparison of XPS with SIMS

### The main XPS advantages:

1. More developed chemical state analysis capability.
2. Some what more accurate elemental analysis.
3. Far fewer problems with induced sample damage and charging effects for insulators.
4. Least destructive to the sample (SIMS is an intrinsically destructive technique).

### The advantages of SIMS:

1. Sensitivity in surface characterization.
2. Good at trace analysis, (not for XPS).
3. Probes even less deeply at the surface.
4. Spatial resolution is better.
5. Detects H.

### Conclusion:

The two techniques are very complementary in respect of quantification, structural insight, sampling depth and in situations such as filler surface analysis, where layered systems involving both organic and inorganic components are involved, SIMS is a valuable adjunct to XPS.

## 2.5. Application of Fouriour Transform Infrared Spectroscopy

### Fundamentals of vibrational spectroscopy

Vibrational spectroscopy is concerned with the detection of transition between energy levels in molecules which result from vibrations of the interatomic bonds. The vibrational frequencies are shown to be characteristic of particular functional groups in molecules. They are sensitizes to the molecular environment, chain conformations and morphology and so afford a useful method for polymer analysis.

When molecular vibrations result in a change in the bond dipole moment, as a consequence of change in the electron distribution in the bond, it is possible to stimulate transitions between energy levels by interaction with EM radiation of the appropriate frequency. In effect , when the vibrating dipole is in phase with the electric vector of the incident radiation the vibrations are enhanced and there is transfer of energy from the incident radiation to the molecule. It is the detection of this energy absorption which constitutes IR spectroscopy. In practice, the spectral transitions are detected by scanning through the frequency whilst continuously monitoring the transmitted light intensity. The energies of molecular vibrations of interest for analytical work correspond to EM wavelengths in the range 2.5-25um or ,when expressed conventionally in terms of wavenumber, 4000-400cm-1 (frequency in wavenumber (cm-1)=10000/wavelength in um). Some spectrometers operate in the near infra-red region (NIH) 0.7 to 2.5um (4000 to 1400 cm-1) and others in the far infra-red (FIR) 50 to 800 um (200 to 12 cm-1).

## Special features and applications of DRIFT

1. Giving intimate structural detail of the molecules under examination.
2. No need of elaborate preparation (grinding etc.) which can modify the chemical nature of the material.
3. The spectra of uncoated filler is able to be subtracted from that of the coated specimen, creating a difference spectra of the surface coating. The accumulation of data from multiple scans permits spectra to build up from small concentrations, for example, monolayers, of surface coatings

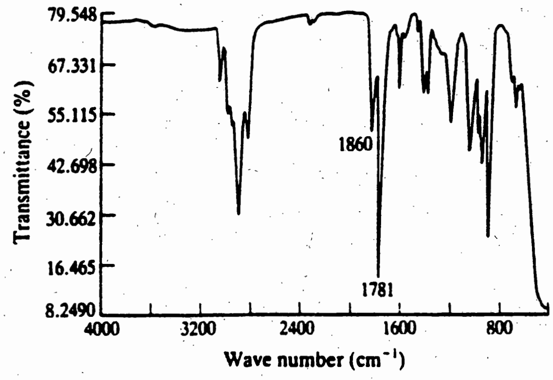

**Figure** Transmission infrared spectrum of maleinized polybutadiene.

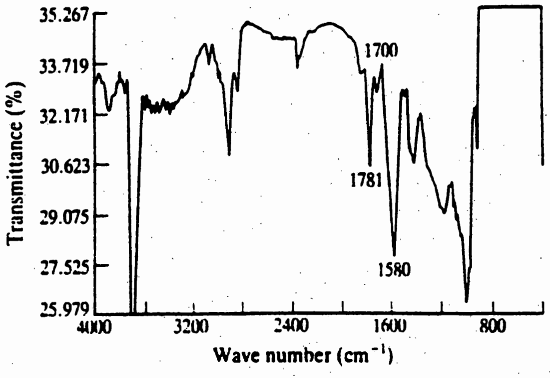

**Figure** DRIFT infrared difference spectrum of magnesium hydroxide subtracted from magnesium hydroxide coated with maleinized polybutadiene.

# 3. Transmission Electron Microscopy and its Applications

## 3.1. Introduction

Transmission Electron Microscopy (TEM) has, in three decades time, become a mainstay in the repertoire of characterization techniques for materials scientists. TEM's strong cards are its high lateral spatial resolution (better than 0.2 nm "point-to -point" on some instruments. Although such high resolution examination is seldom possible with polymers, it is possible to obtain information within the range 1-100 nm with varying degrees of difficulty.) and its capability to provide both image and diffraction information from a single sample. In addition, the highly energetic beam of electrons used in TEM interacts with sample matter to produce characteristic radiation and particles; these signals often are measures to provide materials characterization using EDS, EELS, EXELFS, backscattered and secondary electron imaging, to name a few possible techniques.

There are two types of TEM, the conventional TEM (CTEM) and the scanning TEM (STEM). CTEM is mainly discussed here and the STEM will be dealt with briefly later.

## 3.2. Basic principles

In TEM, a focused electron beam is incident on a thin (less than 200 nm) sample. The signal in TEM is obtained from both undeflected and deflected electrons that penetrate the sample thickness. A series of magnetic lenses at and below the sample position are responsible for delivering the signal to a detector, usually a fluorescent screen, a film plate, or a video camera. Accompanying this signal transmission is a magnification of the spatial information in the signal by as little as 50 times to as much as a factor of 1,000,000. This remarkable magnification range is facilitated by the small wavelenth of the incident electrons, and is the key to the unique capabilities associated with TEM analysis.

### Layout of the TEM

The CTEM can be likened to the light microscope operated in transmission, consisting of a source (of electron instead of light), a condenser system, a specimen stage, an objective lens and a projector system. A major difference follows from the ready interaction of electron with matter (including gas) for in the TEM the path from source to detector must be in vacuum so that the only origin of scattering is the specimen.

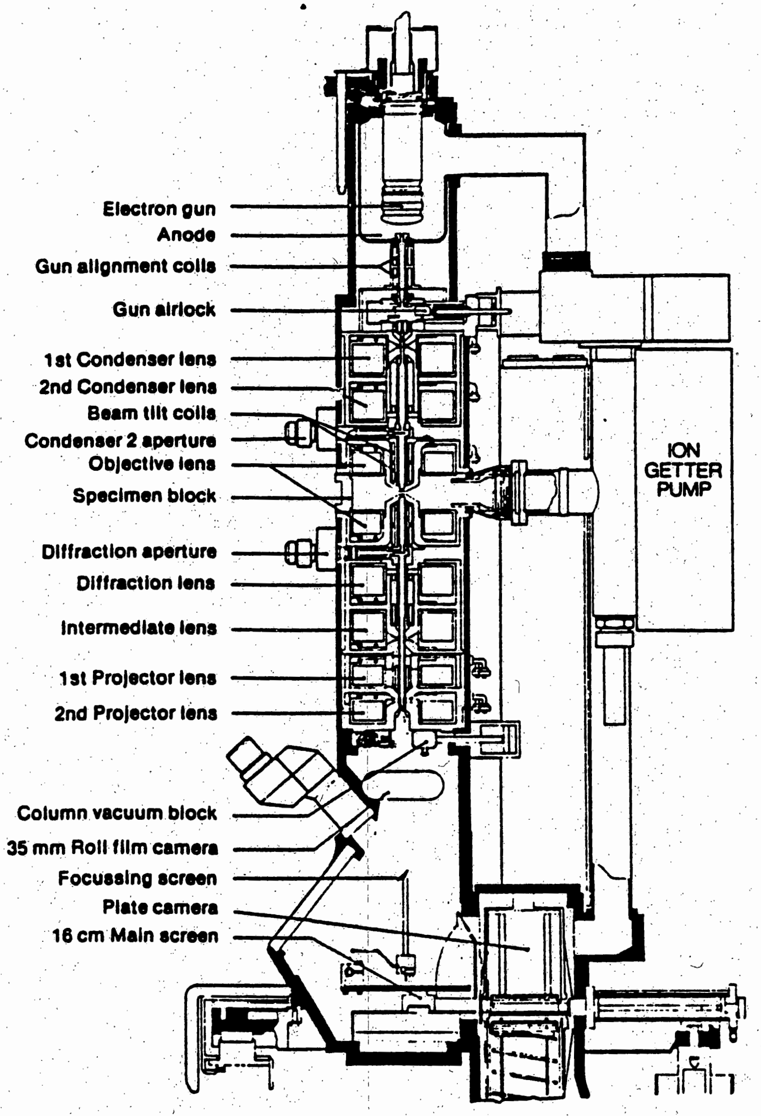

**Figure** Schematic diagram of a TEM instrument, showing the location of a thin sample and the principal lenses within a TEM column.

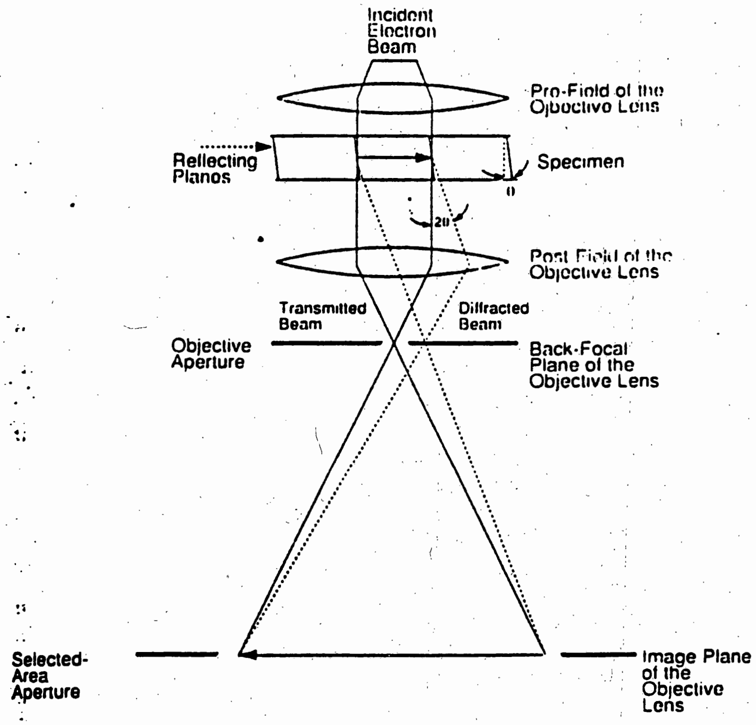

**Figure** Schematic representation for the ray paths of both unscattered and scattered electrons beneath the sample.

## Resolution

In light microscopy the ultimate resolution is diffraction-limited and is of the order of the wavelength, i.e. around 500nm for visible light. Thus to form images of detail smaller than this, radiation of smaller wavelength is required and this led to the development of the TEM. Moving electron have a wave nature, and after 100kv acceleration the wavelength is approximately 0.0037nm. This is below atomic dimensions, but resolution now becomes limited by the aberration of the lenses and/or by the deficiencies of the detection system (e.g. the phosphor screen on which the image is displayed). Higher voltages produce shorter wavelengths and although this in itself is not of any benefit when resolution is determined by other factors, it permits the construction of lenses with slightly better image forming characteristics.

A second motive for building TEM with higher voltages is that electron penetration increases, permitting the use of thicker specimen.

High lateral spatial resolution is maintained via the use of extremely thin samples. In most TEM experiments, samples are thinned usually to less than 200nm. For most materials this insures relatively few scattering events as each electron traverses the sample. Not only does this limit spreading of the probe, but much of the coherency of the incident sources is also retained.

## Sensitivity

TEM has no inherent ability to distinguish atomic species. Nonetheless, electron scattering is exceedingly sensitive to the target element. Heavy atoms having large, positively charged nuclei, scatter electrons more effectively and to higher angles of deflection, than do light atoms.

## Electron optics

At the top of the TEM column is an electron source or gun. An electrostatic lens is used to accelerate electron emitted by the filament to a high potential (typically 100-1000kv) and to focus the electrons to a cross-over just above the anode (the diameter of the cross-over image can be from 0.5 to 30 um, depending on the type of gun). The electrons at the cross-over image of the filament are delivered to the specimen by the next set of lenses on the column, the condensers.

Most modern TEMs use a two-stage condenser lens system that makes it possible to:

1 produce a highly demagnified image of cross-over at the specimen, such that only a very small sample region is illuminated (typically < 1 um).

2 Focus the beam at "infinity" to produce nearly parallel illumination at the specimen.

The former procedure is the method of choice during operation in the image mode, while the latter condition is desirable for maximizing source coherency in the diffraction mode.

The specimen is immersed in the next lens encountered along the column, the objective lens. The objective lens is a magnetic lens. Instrumental resolution is limited primarily by the spherical aberration of the objective lens.

The final set of magnetic lenses beneath the specimen are jointly referred to as post-specimen lenses. Their primary task is to magnify the signal transferred by the objective lens. Modern instruments typically contain four post-specimen lenses: diffraction, intermediate, projector 1, and projector 2 (in order of appearance below the specimen). They provide a TEM with its tremendous magnification flexibility.

Collectively, the post-specimen lenses serve one of two purposes: they magnify either the diffraction pattern from the sample produced at the back focal plane of the objective lens; or they magnify the image produced at the image plane of the objective lens. These optical planes are illustrated in the electron ray diagram in Figure . By varying the lenses' strength so as to alternate between these two object planes, the post- specimen lenses deliver either a magnified diffraction pattern or a magnified image of the specimen to the detector.

The purpose of apertures is to filter either the source or the transmitted signal. The most important diaphragm is called the objective aperture. This aperture lies in the back focal plane of the objective lens. In this plane the scattered electron waves recombine to form a diffraction pattern. Inserting an aperture in this plane effectively blocks certain scattered waves. Use of a small objective aperture while operating in the image mode, which blocks all diffracted beams (as in this example), can serve to enhance significantly image contrast. Use of a large objective aperture, that allows the passage of many diffracted beams, is the modus operandi for the technique referred to as high-resolution transmission electron microscopy (HRTEM).

## 3.3. TEM operation

### Diffraction mode

TEM offers two methods of specimen observation, diffraction mode and image mode. In diffraction mode, an electron diffraction pattern is obtained on the fluorescent screen, where the post-specimen lenses are set to examine the information in the transmitted signal at the back focal plane of the objective lens.

The diffraction pattern is entirely equivalent to an X-ray diffraction pattern:

A single crystal will produce a spot pattern on the screen, a polycrystal will produce a power or ring pattern (assuming the illuminated area includes a sufficient quantity of crystallites), and a glassy or amorphous material will produce a series of diffuse halos.

### Image mode

The image mode produces an image of the illuminated sample area, as in Figure . The image can contain contrast brought about by several mechanisms: mass contrast, due to spatial separations between distinct atomic constituents; thickness contrast, due to nonuniformity in sample thickness; diffraction contrast, which in the case of crystalline materials results from scattering of the incident electron wave by structural defects; and phase contrasts.

In image mode, the post-specimen lenses are to examine the information in the transmitted signal at the image plane of the objective lens. Here, the scattered electron waves finally recombine, forming an image with recognizable details related to the sample microstructure (or atomic structure).

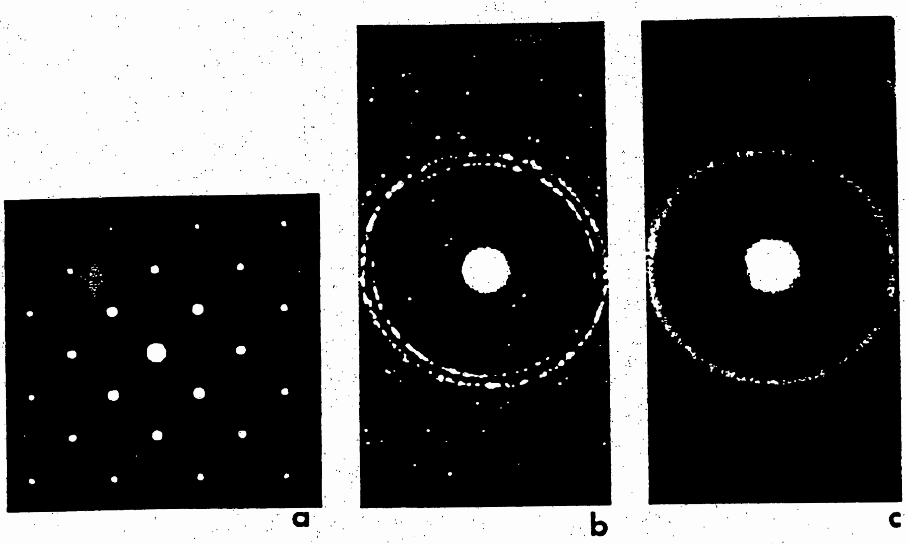

**Figure** (a) Diffraction pattern from a single crystal Fe thin film, oriented with the [001] crystal axis parallel to the incident electron beam direction. (b) Diffraction pattern from a polycrystalline thin film of $Pd_2Si$. (c) Diffraction pattern from the same film as in (c), following irradiation of the film with 400-keV $Kr^+$ ions. See text for discussion (b, c Courtesy of M. Nastasi, Los Alamos National Laboratory)

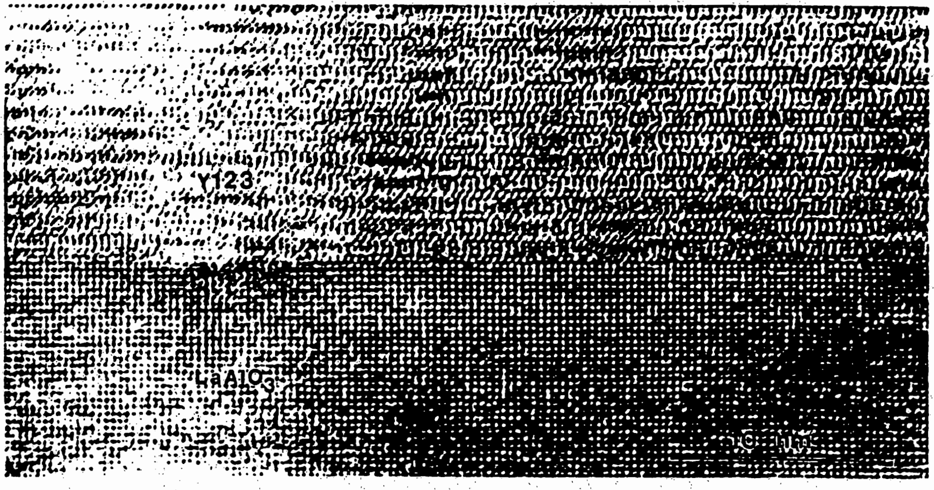

**Figure** High-resolution transmission electron microscopy image of an epitaxial thin film of $Y_1Ba_2Cu_3O_{7-x}$ grown on $LaAlO_3$, shown in cross section. (Courtesy of T. E. Mitchell, Los Alamos National Laboratory)

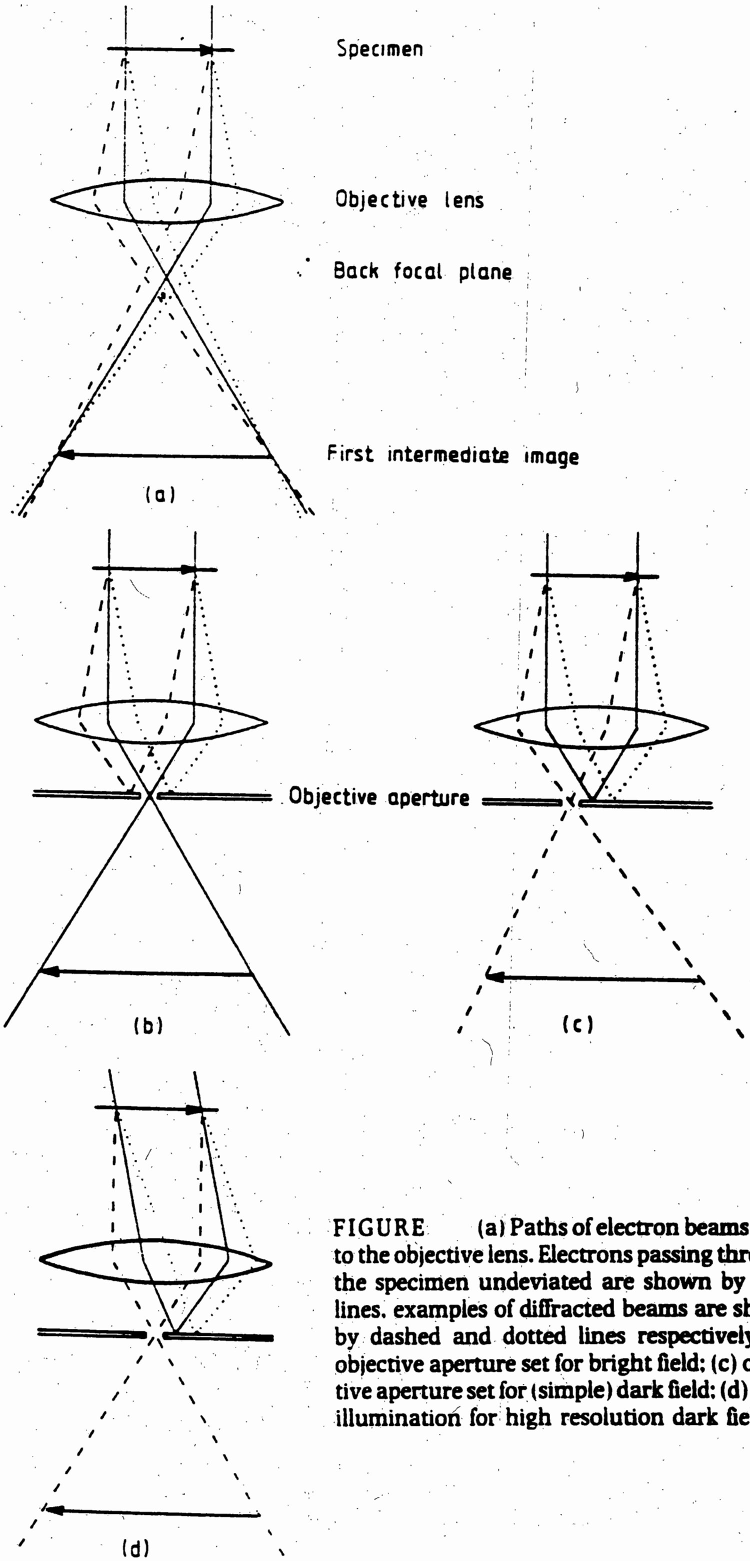

FIGURE (a) Paths of electron beams near to the objective lens. Electrons passing through the specimen undeviated are shown by solid lines. examples of diffracted beams are shown by dashed and dotted lines respectively; (b) objective aperture set for bright field; (c) objective aperture set for (simple) dark field; (d) tilted illumination for high resolution dark field.

### Three primary image modes used in TEM work:

Bright-field microscopy
Dark-field microscopy
High-resolution-electron microscopy

### Specimen preparation

The most difficult, yet at the same time, most important aspect of the TEM technique is the preparation of high-quality thin foils for observation. Sample preparation was divided into two categories, one for thin films and one for bulk materials:

Thin-films, particularly metal layers, were often deposited on substrate and later removed by some sort of technique involving dissolution of the substrate.

Bulk materials were cut and polished into thin slabs, which were then either electropolished or ion-milled.

## 3.4. Applications and examples

Advantages of the TEM:

1. It provides detailed structural information at the level down to atomic dimensions.

2. It can be rapidly adjusted to provide an electron diffraction pattern from a selected area, facilitating investigation of crystal structure and orientation, and enabling particular morphological features to be identified.

Main disadvantage of the TEM:

It can be used only on thin samples, less than 1um thick, and preferably less than 100 nm thick.

FIGURE Bright field image of shadowed multilayered solution-grown polyethylene crystals. A rather heavy shadowing deposit has been used for clarity.

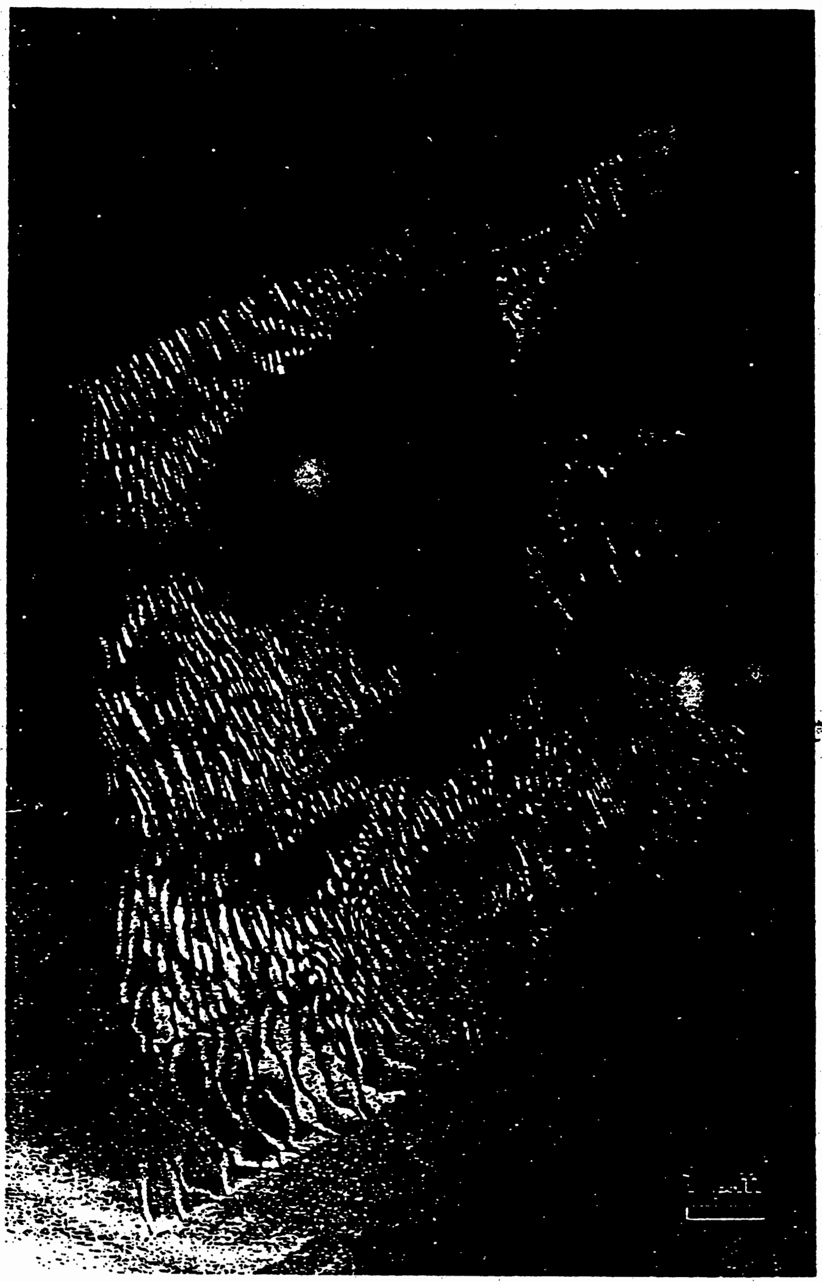

FIGURE Simple dark field image of a multilayered stack of solution grown polyethylene crystals, showing moiré patterns and, near the bottom left-hand corner, a screw dislocation network.

## Applications in surface study:

1. Crazes in thin films.
2. Observe directly an image of the intersurface.

## STEM

In STEM the lenses above the specimen are used to focus the electron beam into a very fine probe at the specimen. The probes scanned across the specimen under the command of scanning coils in the same way as in the SEM. The interaction of the electron beam with the specimen produces various signals, including secondary electrons and X-rays are used to produce images or elemental analysis in the same way as in the SEM, given the appropriate detectors and counters.

During STEM imaging the beam moves rapidly from one position to the next so that destruction of the specimen does not necessarily take place, even the intensity is very high within the prob.

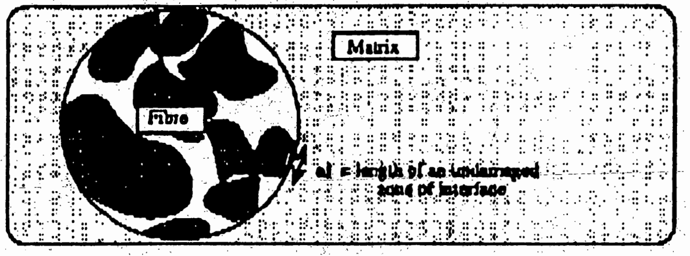

Fig. Microtomed transverse cross-section of a carbon-fibre reinforced composite. Contact index = Σ ai/perimeter.

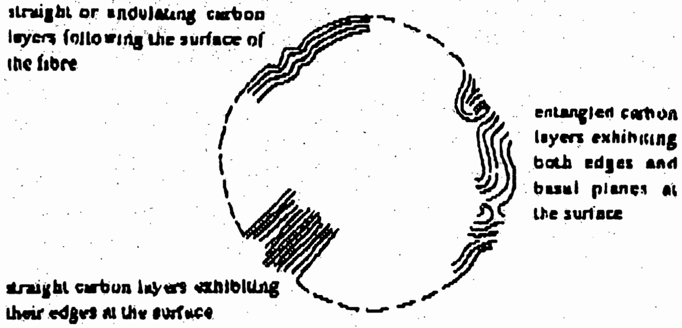

Fig. Arrangements of the carbon layers at the surface of PAN-based carbon fibres.

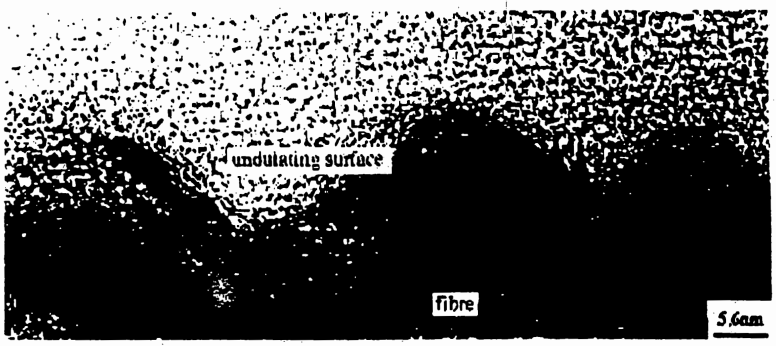

Fig. 3.10 Transverse cross-section of the fiber M40 showing carbon layers parallel to the surface.

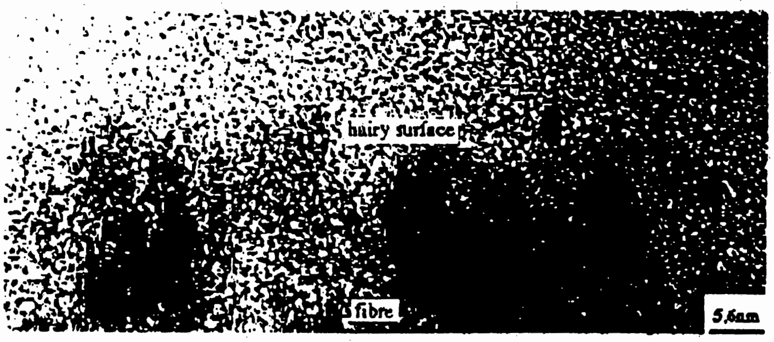

Fig. 3.11 Transverse cross-section of the fiber M40 showing carbon-layer edges at the surface.

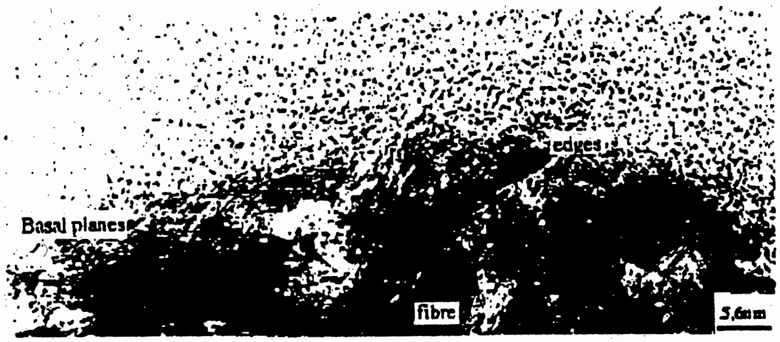

Fig. 3.12 Transverse cross-section of the fiber M40 showing an intermediate surface.

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## Concentrations in sample LEAD-1

| | Compound | Conc. (%) | Compound | Conc. (%) | Compound | Conc. (%) | Compound | Conc. (%) | Compound | Conc. (%) |
|---|---|---|---|---|---|---|---|---|---|---|
| 1 | | | Ca | 1.969 | Zr | 0.427 | Nd | | Hg | |
| 2 | H | | Sc | | Nb | | Pm | | Tl | |
| 3 | He | | Ti | 2.972 | Mo | | Sm | | Pb | 11.802 |
| 4 | Li | | V | | Tc | | Eu | | Bi | |
| 5 | Be | | Cr | 0.095 | Ru | | Gd | | Po | |
| 6 | B | | Mn | 0.11 | Rh | | Tb | | At | |
| 7 | C | | Fe | 11.337 | Pd | | Dy | | Rn | |
| 8 | N | | Co | | Ag | | Ho | | Fr | |
| 9 | O | 38.401 | Ni | 0.022 | Cd | | Er | | Ra | |
| 10 | F | | Cu | 0. | In | | Tm | | Ac | |
| 11 | Ne | | Zn | 1.554 | Sn | | Yb | | Th | |
| 12 | Na | | Ga | | Sb | | Lu | | Pa | |
| 13 | Mg | 0.825 | Ge | | Te | | Hf | | U | |
| 14 | Al | 7.571 | As | 0. | I | | Ta | | Np | |
| 15 | Si | 18.189 | Se | | Xe | | W | | Pu | |
| 16 | P | 0.379 | Br | | Cs | | Re | 0. | | |
| 17 | S | 0.439 | Kr | | Ba | 0.404 | Os | 0. | | |
| 18 | Cl | 0.125 | Rb | 0.546 | La | | Ir | 0. | | |
| 19 | Ar | | Sr | 0.045 | Ce | | Pt | | | |
| 20 | K | 2.789 | Y | | Pr | | Au | | | |

100 %

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**Concentrations in sample LEAD-1**

| | Compound | Conc. (%) | Compound | Conc. (%) | Compound | Conc. (%) | Compound | Conc. (%) | Compound | Conc. (%) |
|---|---|---|---|---|---|---|---|---|---|---|
| 1 | | | CaO | 2.755 | ZrO2 | 0.576 | Nd | | Hg | |
| 2 | H | | Sc | | Nb | | Pm | | Tl | |
| 3 | He | | TiO2 | 4.957 | Mo | | Sm | | PbO | 12.713 |
| 4 | Li | | V | | Tc | | Eu | | Bi | |
| 5 | Be | | Cr2O3 | 0.139 | Ru | | Gd | | Po | |
| 6 | B | | MnO2 | 0.174 | Rh | | Tb | | At | |
| 7 | C | | Fe2O3 | 16.209 | Pd | | Dy | | Rn | |
| 8 | N | | Co | | Ag | | Ho | | Fr | |
| 9 | O | | NiO | 0.027 | Cd | | Er | | Ra | |
| 10 | F | | CuO | F0. | In | | Tm | | Ac | |
| 11 | Ne | | ZnO | 1.935 | Sn | | Yb | | Th | |
| 12 | Na | | Ga | | Sb | | Lu | | Pa | |
| 13 | MgO | 1.369 | Ge | | Te | | Hf | | U | |
| 14 | Al2O3 | 14.305 | As2O3 | F0. | I | | Ta | | Np | |
| 15 | SiO2 | 38.913 | Se | | Xe | | W | | Pu | |
| 16 | P2O5 | 0.869 | Br | | Cs | | Re | F0. | | |
| 17 | SO3 | 1.097 | Kr | | BaO | 0.451 | Os | F0. | | |
| 18 | Cl | 0.125 | Rb | 0.546 | La | | Ir | F0. | | |
| 19 | Ar | | SrO | 0.053 | Ce | | Pt | | | |
| 20 | K | 2.789 | Y | | Pr | | Au | | | |

100%

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## Concentrations in sample LEAD-1

| | Compound | Conc. (%) | Compound | Conc. (%) | Compound | Conc. (%) | Compound | Conc. (%) | Compound | Conc. (%) |
|---|---|---|---|---|---|---|---|---|---|---|
| 1 | | | Ca | 1.128 | Zr | 0.244 | Nd | | Hg | |
| 2 | H | | Sc | | Nb | | Pm | | Tl | |
| 3 | He | | Ti | 1.703 | Mo | | Sm | | Pb | 6.763 |
| 4 | Li | | V | | Tc | | Eu | | Bi | |
| 5 | Be | | Cr | 0.054 | Ru | | Gd | | Po | |
| 6 | B | | Mn | 0.063 | Rh | | Tb | | At | |
| 7 | C | | Fe | 6.497 | Pd | | Dy | | Rn | |
| 8 | N | | Co | | Ag | | Ho | | Fr | |
| 9 | O | 22.005 | Ni | 0.012 | Cd | | Er | | Ra | |
| 10 | F | | Cu | 0. | In | | Tm | | Ac | |
| 11 | Ne | | Zn | 0.891 | Sn | | Yb | | Th | |
| 12 | Na | | Ga | | Sb | | Lu | | Pa | |
| 13 | Mg | 0.473 | Ge | | Te | | Hf | | U | |
| 14 | Al | 4.338 | As | 0. | I | | Ta | | Np | |
| 15 | Si | 10.423 | Se | | Xe | | W | | Pu | |
| 16 | P | 0.217 | Br | | Cs | | Re | 0. | | |
| 17 | S | 0.252 | Kr | | Ba | 0.231 | Os | 0. | | |
| 18 | Cl | 0.072 | Rb | 0.313 | La | | Ir | 0. | | |
| 19 | Ar | | Sr | 0.026 | Ce | | Pt | | | |
| 20 | K | 1.598 | Y | | Pr | | Au | | | |

57.3°

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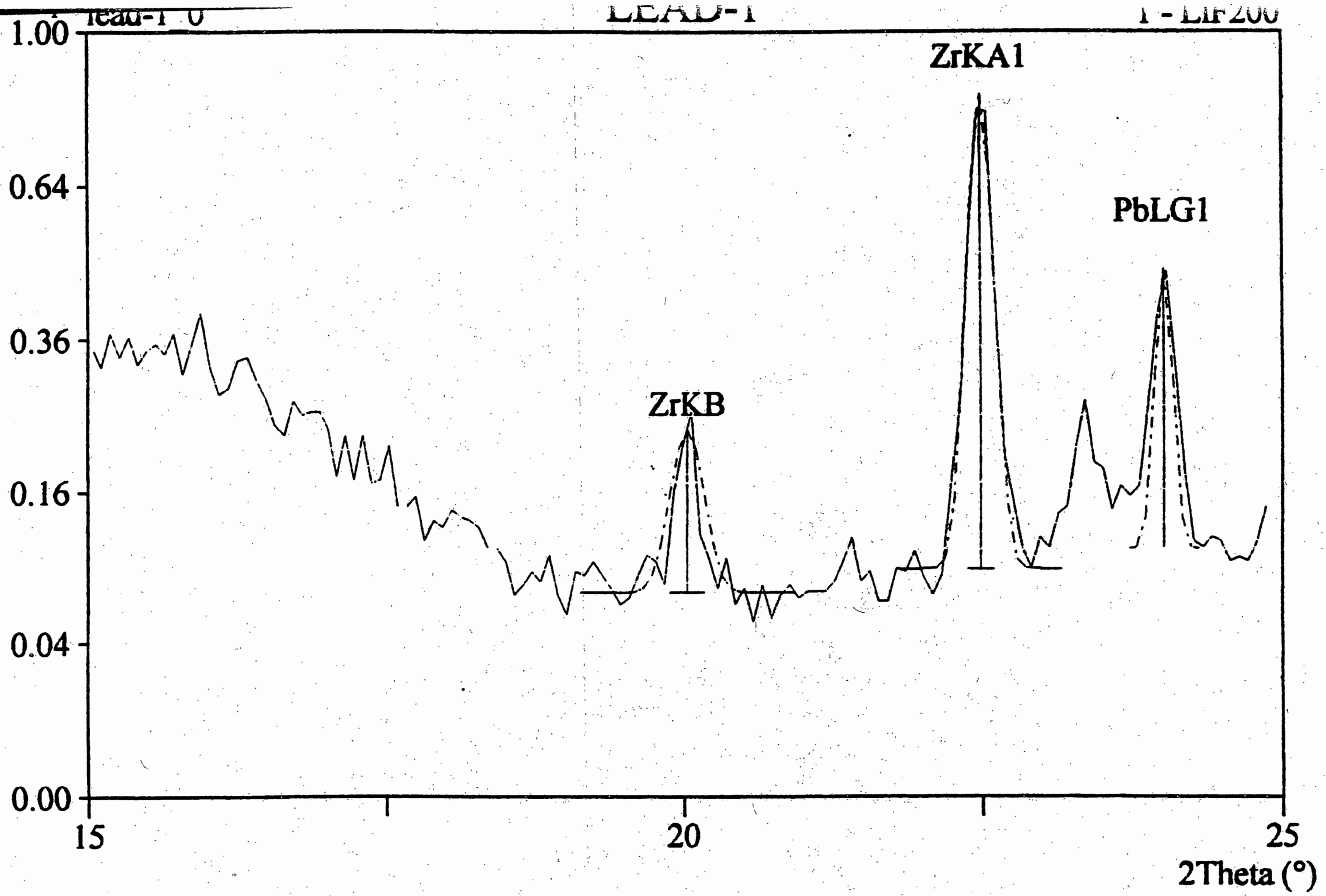
## Concentrations in sample LEAD-1

| | Compound | Conc. (%) | Compound | Conc. (%) | Compound | Conc. (%) | Compound | Conc. (%) | Compound | Conc. (%) |
|---|---|---|---|---|---|---|---|---|---|---|
| 1 | | | CaO | 1.579 | ZrO2 | 0.33 | Nd | | Hg | |
| 2 | H | | Sc | | Nb | | Pm | | Tl | |
| 3 | He | | TiO2 | 2.841 | Mo | | Sm | | PbO | 7.285 |
| 4 | Li | | V | | Tc | | Eu | | Bi | |
| 5 | Be | | Cr2O3 | 0.079 | Ru | | Gd | | Po | |
| 6 | B | | MnO2 | 0.1 | Rh | | Tb | | At | |
| 7 | C | | Fe2O3 | 9.288 | Pd | | Dy | | Rn | |
| 8 | N | | Co | | Ag | | Ho | | Fr | |
| 9 | O | | NiO | 0.016 | Cd | | Er | | Ra | |
| 10 | F | | CuO | F0. | In | | Tm | | Ac | |
| 11 | Ne | | ZnO | 1.109 | Sn | | Yb | | Th | |
| 12 | Na | | Ga | | Sb | | Lu | | Pa | |
| 13 | MgO | 0.784 | Ge | | Te | | Hf | | U | |
| 14 | Al2O3 | 8.197 | As2O3 | F0. | I | | Ta | | Np | |
| 15 | SiO2 | 22.298 | Se | | Xe | | W | | Pu | |
| 16 | P2O5 | 0.498 | Br | | Cs | | Re | F0. | | |
| 17 | SO3 | 0.628 | Kr | | BaO | 0.258 | Os | F0. | | |
| 18 | Cl | 0.072 | Rb | 0.313 | La | | Ir | F0. | | |
| 19 | Ar | | SrO | 0.03 | Ce | | Pt | | | |
| 20 | K | 1.598 | Y | | Pr | | Au | | | |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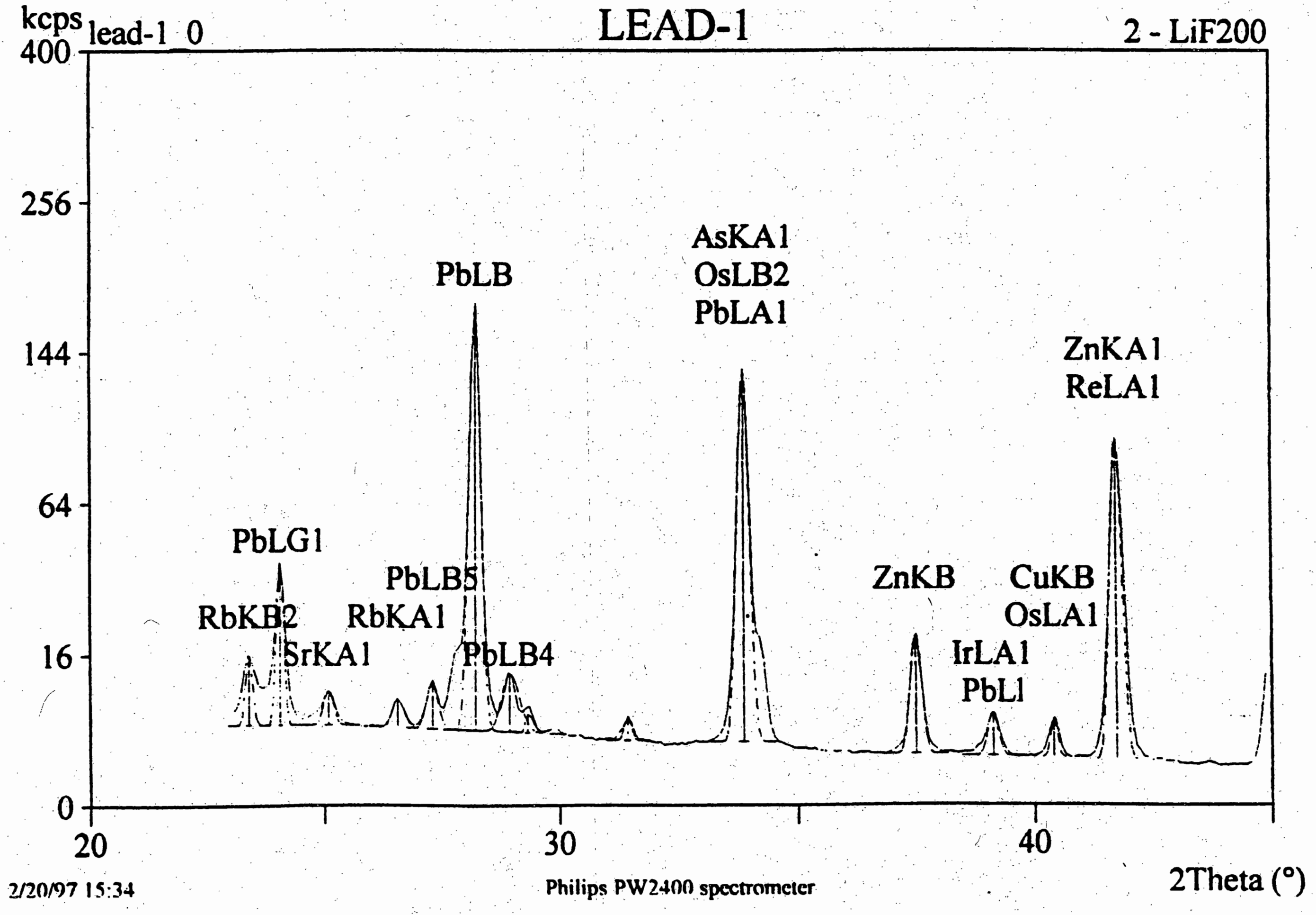
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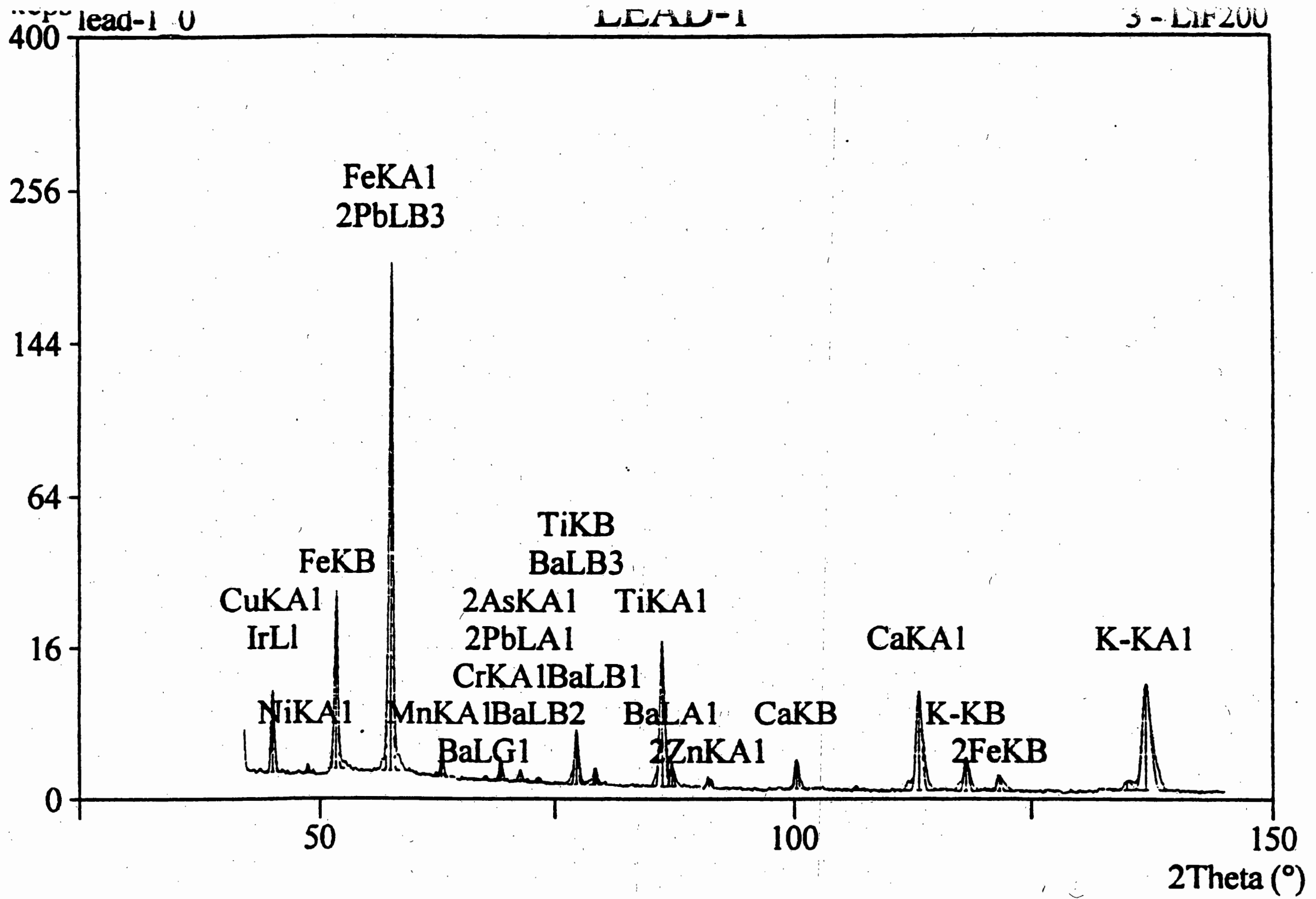
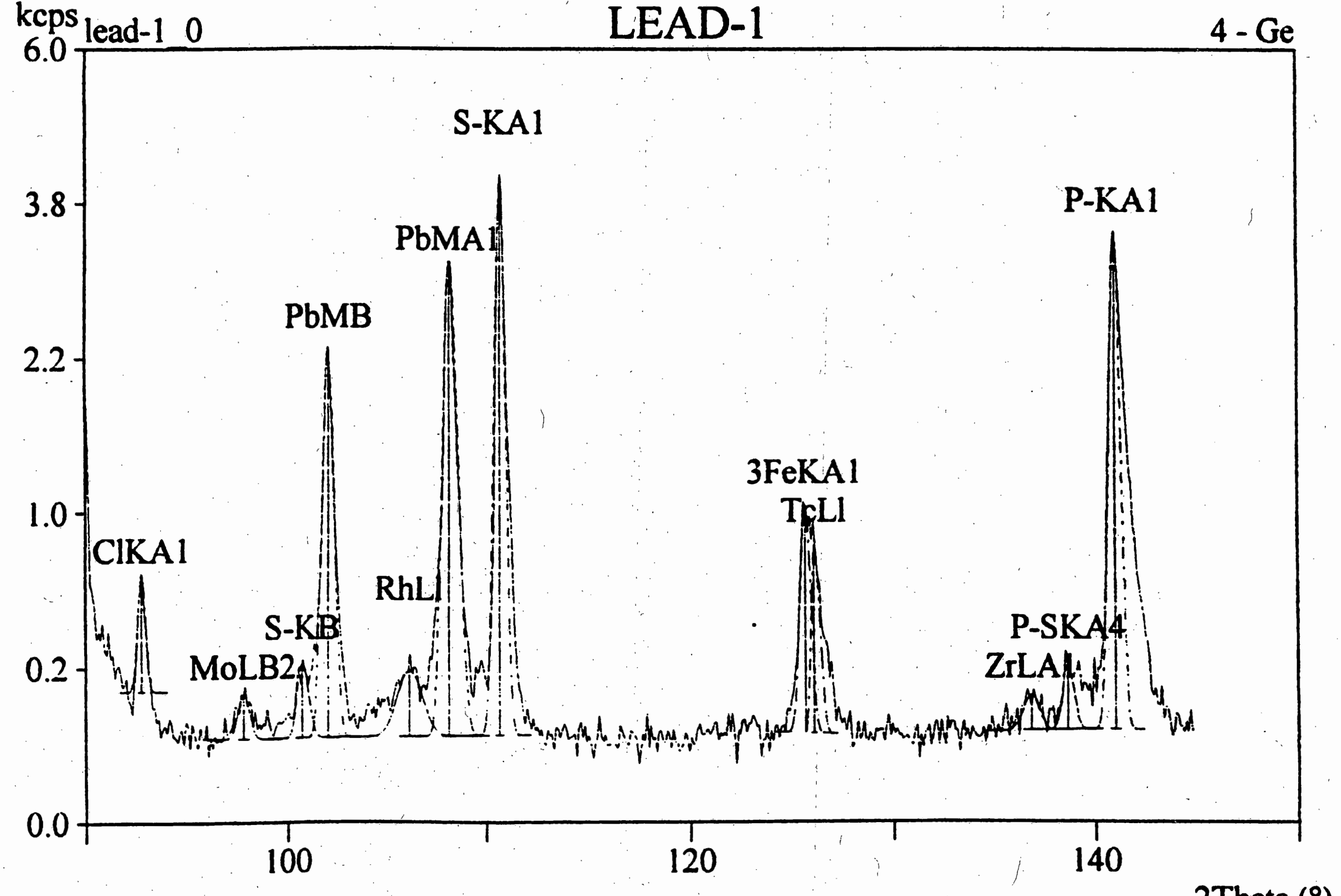
Philips PW2400 spectrometer





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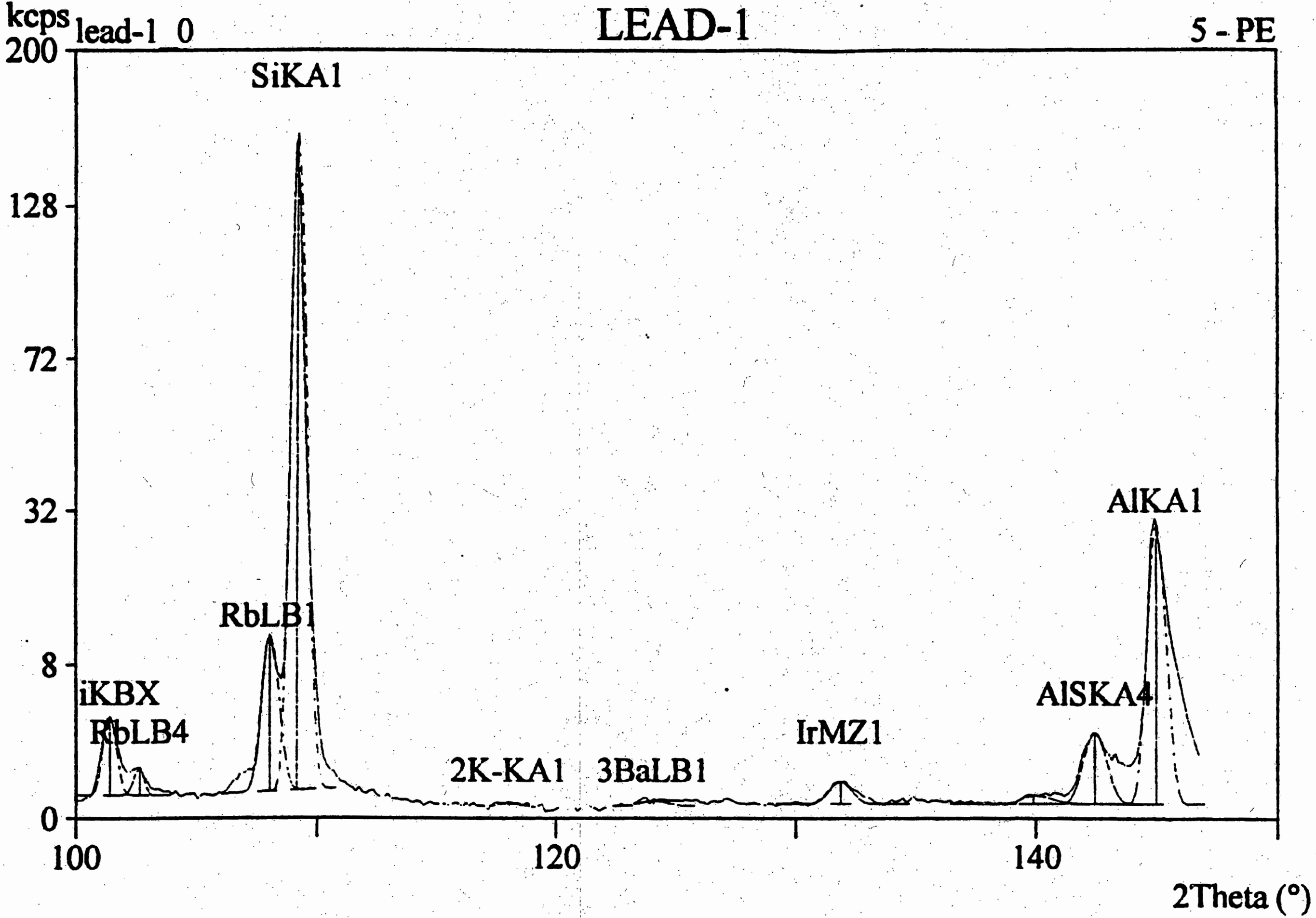
Philips PW2400 spectrometer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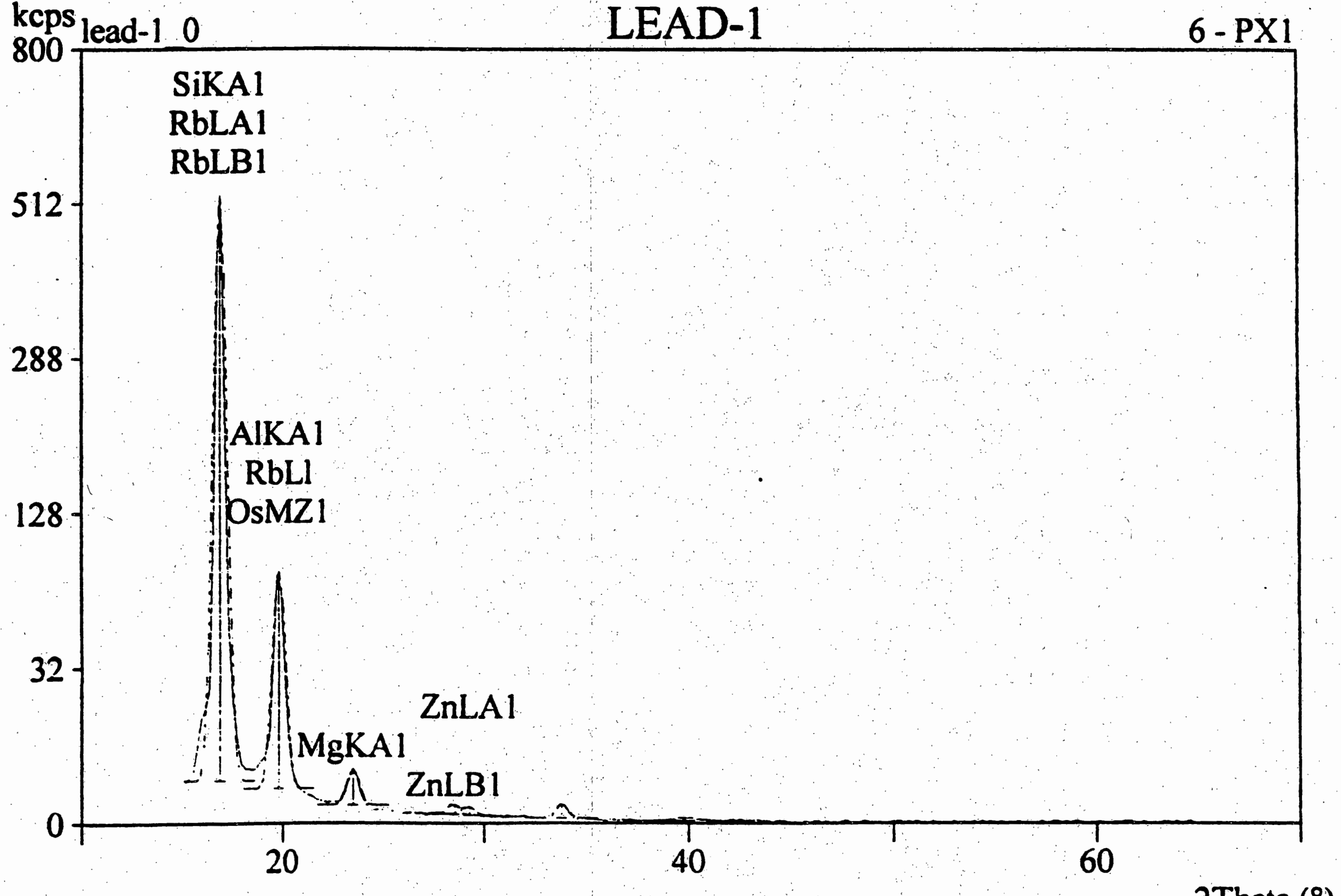
2Theta (°)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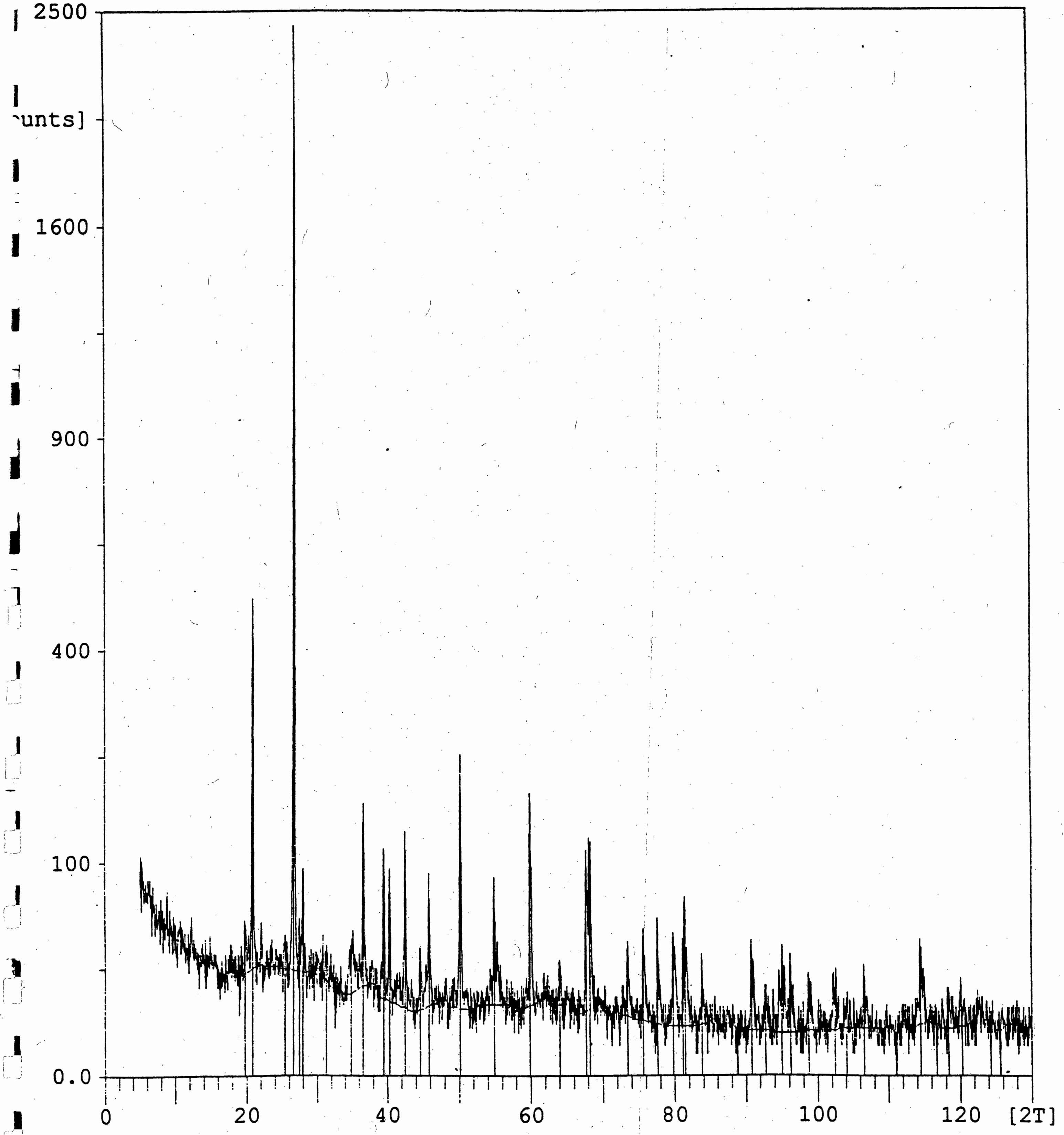

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ilips Analytical                                PC-APD, Diffraction software

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 [°2θ]     α1 [Å]      α2 [Å]   [°2θ]  [counts] [counts]    [%]
-------  ----------  ---------- -------  ------- -------- -------  --------
 68.235   1.37331     1.37673   0.250       69       10      2.9       1.72
 73.450   1.28815     1.29135   0.150       28        7      1.2       1.04
 75.620   1.25648     1.25961   0.200       30        7      1.3       1.74
 77.635   1.22883     1.23188   0.150       49        6      2.0       0.91
 79.840   1.20034     1.20333   0.150       36        5      1.5       0.92
 81.135   1.18442     1.18736   0.150       36        5      1.5       1.49
 81.445   1.18070     1.18363   0.150       61        5      2.5       1.02
 83.810   1.15329     1.15616   0.150       25        6      1.0       1.10
 90.810   1.08172     1.08441   0.150       36        4      1.5       1.70
 92.780   1.06385     1.06649   0.300       11        4      0.5       0.80
 95.010   1.04468     1.04728   0.800       14        4      0.6       1.76
 96.240   1.03457     1.03714   0.150       27        4      1.1       1.76
 98.745   1.01492     1.01744   0.150       17        4      0.7       1.07
102.450   0.98803     0.99049   0.800       11        4      0.5       1.53
104.105   0.97680     0.97923   0.500        7        5      0.3       0.76
106.585   0.96081     0.96320   0.150       22        5      0.9       0.90
114.475   0.91600     0.91827   0.150       35        6      1.4       1.10
118.390   0.89681     0.89904   0.300       12        5      0.5       1.15
 20.280   0.88819     0.89040   0.600        5        5      0.2       0.93

===========================================================================
```

| NAME | FORMULA |
|---|---|
| 1. Quartz Low | $SiO_2$ |
| 2. Quartz Syn | $SiO_2$ |
| 3. Osbornite, Syn | $TiN$ |
| 4. Chromium Titanium Hydri | $Cr_{1.8}TiH_{5.3}$ |
| 5. Titanium Oxide | $TiO_2$ |
| 6. Iron Carbide | $Fe_3C$ |
| 7. Despujolsite | $Ca_3Mn(SO_4)_2(OH)_6 . 3H_2O$ |
| 8. Wuelfingite, Syn | $Zn(OH)_2$ |
| 9. Barium Zinc Phosphate | $Ba_2Zn(PO_4)_2$ |
| 10. Potassium Barium Chromic | $K_2Ba(CrO_4)_2$ |
| 11. Auglesite, Syn | $PbSO_4$ |
| 12. Lithium Titanium Phosphate | $LiTi_2(PO_4)_3$ |
| 13. Calcium Zirconium Oxide | $Ca_6Zr_{19}O_{44}$ |
| 14. Gismondite | $(Ca,K_2) Al_2Si_2O_8. 4H_2O$ |
| 15. Sanidine, disordered | $K(Si_3Al)O_8$ |
| 16. Anorthite, ordered | $CaAl_2Si_2O_8$ |
| 17. Lead Styphnate (basic) | $C_{12}H_{10}N_6O_{31}Pb_{13}$ |
| 18. Sodium Zinc Cyanide | $Na_2Zn(CN)_4$ |
| 19. Calcium Mangauese Oxide | $Ca_2Mn_3O_8$ |
| 20. Biotite-1\1TM\RG,Ferri | $K(Mg,Fe)_3(Si_3Al)O_{10}(OH,F)_2$ |
| 21. Giniite | $FeFe_4(PO_4)(OH)_2. 2H_2O$ |
| 22. Lead Phosphate | $Pb_3(PO_4)_2$ |

# RESULTS III

# THE DEVELOPMENT OF DURABLE ASPHALT PAVEMENTS USING FISHING NETS AS REINFORCEMENT

**by**

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**ENE 700B - 208**

**A Master's Project**

**Submitted to Dr. Mohamed E. Labib**

**November 17, 1994**

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# 1.0 INTRODUCTION

## 1.1 FISHING NET WASTE

Discarded fishing nets and ropes remain to be a major marine pollutant problem. Fishing nets discarded at sea entangle and kill marine mammals, birds and fish, disable ships, and aesthetically degrade beaches. Worldwide it is estimated that 287 million pounds of netting are discarded annually, and at least 2 million pounds in the United States alone. Concerns for the growing use and disposal of plastic items at sea led to the implementation of Annex V of MARPOL at the end of 1988. This treaty and resultant US law prohibited the disposal of any plastic item at sea and mandated that these items be brought ashore for disposal[1].

Although these restrictions have substantially reduced the problem of marine debris and the associated environmental hazards, another severe problem has resulted: the land based disposal of used and obsolete fishing nets. The fishing net industry currently disposes of their unwanted fishing gear in landfills or stock it in storage area. A limited amount of nylon gill netting is baled and shipped overseas for reprocessing. In other instances some materials are incinerated. However, problems have been experienced due to the insufficient capacity of the landfills and the air pollution associated with incinerators.

Many efforts have been made to resolve these problems. One study demonstrated that fishing nets and ropes could be melt recycled. However, from an economic standpoint, considerable questions remain due to the high costs of sorting , cleaning, and preparing for the melting process. In other work it was found that it may be possible to use degradable polymer material in fishing nets and other fishing gear[2]. This could partially alleviate the burden of fishing net wastes in the future, but research in this field is still in its infancy.

## 1.2 THE USE OF FIBERS IN ASPHALT

Legislation has been enacted by several states in recent years to either mandate the usage of some waste materials, such as fishing gear, or to examine the feasibility of such usage. About 450 million megagrams of hot mix asphalt (HMA) is produced in the United States alone at a cost of about $12 billion. The HMA industry has been pressured in recent years to incorporate a wide range of waste materials into hot mix asphalt pavements[3].

The deterioration of asphalt concrete pavements due to loading stresses and environmental exposure has warranted the development of new methods for improving pavement properties and extending pavement life. Some investigators have found the most practical way this can be achieved is by incorporating fibers into asphalt, thus forming a composite. The impact of fiber reinforced asphalt has been recognized for several years. Earlier, and now abandoned, technology was based on using asbestos fibers as reinforcement to make airport runway pavement with high performance[4]. Another study, *Polyester Fibers In Asphalt Paving Mixtures*, prepared by Clemson University, showed that the addition of virgin polyester fibers into asphalt significantly improved pavement properties. In addition, eight more studies conducted by the Departments of Transportation of Delaware and Oregon showed that adding fibers to asphalt mixes improved pavement performance. The research indicates that the fiber reinforced asphalt is superior to conventional mixes for : overlays (maintenance), bridge membranes, and composite and multi-course asphalt applications[5].

## 1.3 PURPOSE OF STUDY

With the need to recycle fishing nets and the knowledge that fibers may improve asphalt pavement properties, we performed a study to test the development of durable pavement materials using shredded fishing nets as a source for reinforcing fibers. The novel product would be ideal for use in the transportation sector near disposal centers for fishing nets. The product would solve the problem of marine waste disposal while potentially extending the life and improving the properties of asphalt pavements.

The purpose of this limited study was to determine how different types and amounts of fishing net fibers affect the characteristics of asphalt pavement. The study also allowed for the identification of many important engineering concerns which must be addressed in future, and more extensive, studies in this area. This report is an overview of the study which was conducted.

## 2.0 MATERIALS & EXPERIMENTAL METHODS

### 2.1 THE FIBERS

Two types of fibers were obtained from the New Jersey Marine Science Consortium in Fort Hancock, New Jersey: a monofiliment gill net and a trawl net. The nets were shredded using a shredding machine and the resulting fibers had the following characteristics:

The trawl net is made of a red nylon material. The shredded fibers are curly and range from 1/4" to 1" in length. Original intact net is made up of three strands spiraled together to make up a single strand of about 1/16" in diameter. Each of the three strands is made up of about 300 individual fibers each. These single fibers are very fine, less than 1/100" in diameter. Shredded fibers are present in all possible stages, from single fibers to fully intact strands some even maintaining the knots present in the original netting. The nylon fibers tend to be strong and tough with very high tensile strengths (50,000 psi). Nylon has a melting point of 250°C and the fibers tend to absorb some of the asphalt cement.

The monofiliment trawl net is made of a polypropylene material. The shredded fibers are straight and range from 1/4" to 1" in length. The fibers are present in bunches of one to four and are each about 1/64" (0.5 mm) in diameter. The fibers are of one color each, with each being one of the following colors: green, blue, orange, red, brown, or clear. Polypropylene rope fibers also have high tensile strengths (59,000 psi). They have a softening point of 150°C and will not absorb asphalt cement.

Knots still intact in either of the nets are considerably strong and will pose no physical problems. Minor contaminants and mixed coloration of these waste materials pose difficulties for many potential applications but show no significant adverse effects on the quality of fiber reinforced asphalt. Therefore, it is not necessary to sort or wash the nets prior to their use.

## 2.2 THE ASPHALT CEMENT & AGGREGATE

The binder used in the preparation of test specimens throughout the experiment was AC-20 asphalt cement. Through previous testing (ASTM D 70) the specific gravity of the asphalt cement was determined to be 1.029 . The amount of asphalt used in each test specimen was kept constant at 7% by weight of total aggregate.

The aggregate mixture used was made up of 83.3 % crushed rock and 16.7% sand. The gradation was chosen to meet ASTM standard specifications. A sieve analysis of the gradation was performed (ASTM C 136) to verify this. The results of this analysis are presented in TABLE 1. The gradation will remain constant for this limited experiment to allow for easy comparison of the results. Through previous testing (ASTM C 127/128) the average bulk specific gravity of the total aggregate was determined to be 2.80 .

**TABLE 1**

**SIEVE ANALYSIS OF AGGREGATE**

| SIEVE # | % PASSING | ACCEPTABLE % RANGE |
|---|---|---|
| 3/4" | 98.88 | 97 - 100 |
| 3/8" | 75.98 | 60 - 90 |
| #4 | 53.84 | 50 - 79 |
| #10 | 41.58 | 36 - 67 |
| #40 | 21.15 | 17 - 44 |
| #70 | 9.53 | 9 - 29 |
| #200 | 4.0 | 3 - 8 |

## 2.3.0 MARSHALL TESTING

### 2.3.1 BACKGROUND

A number of different methods have been developed for the design and testing of hot mix asphalt paving mixtures. Two of these methods, the Marshall and the Hveen, are the most commonly used and have been successful for a number of years. Although each method has its merits, the Marshall is the most versatile and has become the most widely used in the United States for highway type pavements. The two principal features of the Marshall test are a density-voids analysis and a stability-flow test of compacted test specimens. The American Society of Testing Materials has adopted the standardized Marshall method as test number ASTM D 1559 : *Resistance of Plastic Flow of Bituminous Mixtures Using Marshall Apparatus.*

### 2.3.2 FIBER INTRODUCTION

The process of fiber introduction into the mixture is not specified by ASTM and is of great importance. There are two potential method for the introduction of the fibers: the wet process and the dry process. The wet process blends the fibers with the asphalt cement prior to incorporating the binder into the mixture. The dry process mixes the fiber with the aggregate before the addition of he asphalt cement. For this limited study the dry process was chosen as the singular method for a number of reasons. Experimentally the dry process is the easiest to perform and allows for the best fiber distribution in the mix. Also, because the fibers used do not melt in the asphalt cement there are no apparent special benefits to the wet process. In addition, the field work done on fiber reinforced asphalt has generally used the dry process[6,7], possibly due to the production problems of introducing the fibers directly into the asphalt cement. Another reason for using the dry process is that it minimizes the major problem of clumping or balling together of fibers in the mix.

For fiber reinforced asphalt to be used extensively an effective method for the introduction of fibers must be found. This problem has plagued much of the previous work using fibers in asphalt pavements. Fibers and fiber clumps attract asphalt cement faster than the aggregates. The result is frequent clumping of the fibers and inadequate aggregate coating. These clumps of fibers reduce the strength of the material and may also mar the surface of the finished pavement. Early research specified longer mix times to remedy the problem. But over mixing tends to cause aggregate separation. Such a mix has low stability qualities because the interlocking friction between the aggregates does not reach its full potential. Studies have been undertaken to remedy this problem, usually involving a separate feed for the introduction of the fibers simultaneously with the aggregate[6,7].

### 2.3.3 SPECIMEN PREPARATION

The first step in the preparation of specimens is the introduction of the fibers. As previously discussed the fibers are added directly to the dry aggregate. The pre weighed fibers are added to the pre weighed aggregate and mixed thoroughly. The mixing is continued until the fibers are evenly distributed throughout the mixture.

The next step is the heating of the materials. The aggregate-fiber mixture is heated in an oven at 150°C . The asphalt cement was heated in a heating bowl at a temperature of about 200°C . The materials are heated for at least one hour before being used. The asphalt is then added to the heated aggregate-fiber mixture and mixed vigorously until a well coated homogeneous mixture results.

The entire batch is then placed in a preheated (150°C) compaction mold. This mold consists of a base plate, a forming mold, and a collar extension secured to a compaction pedestal. The mold cylinders are 4 inches in diameter by three inches in height. After the mixture is spaded vigorously in the mold it is ready for compaction. The compaction hammer consists of a flat, circular tamping face and a 10 pound sliding weight with a free fall of 18 inches. 75 blows are applied to each side of the specimen. The specimen is then allowed to cool at least one hour in the mold before being removed from the mold with an extrusion jack. After sitting overnight the specimens are then ready to be tested.

### 2.3.4 SPECIMEN TESTING

First the height and the weight of the samples are recorded. The bulk specific gravity is then determined according to the designation ASTM D 1188 : *Bulk Specific Gravity of Compacted Bituminous Mixtures Using Paraffin Coated Specimens.* In this procedure the specimen is coated with wax, re weighed, and then suspended in water in order to determine its volume. Using this data the bulk specific gravity of each specimen can be calculated and a density-voids analysis can be performed.

After the determination of the bulk specific gravity the stability and flow tests are performed. The testing apparatus consists of a compression testing device fitted with a breaking head. The breaking head consists of an upper and a lower cylindrical segment each having an inside radius of curvature of 2 inches. The testing device is equipped with load and deformation indicating devices which are recorded by computer.

Each cooled specimen is then immersed in a water bath at 60°C for 30 minutes prior to testing. The specimen is then removed and immediately placed in the lower segment of the breaking head. The upper segment is placed on top of the specimen and the complete assembly is put into place on the testing machine. A load is then applied to the specimen by means of a constant rate of movement of 2 inches per minute applied by the testing machine head until the maximum load is reached. The stability and flow for each specimen is recorded by computer throughout the test.

# 3.0 RESULTS & DISCUSSION

## 3.1 GENERAL INFORMATION

The following results are based on a laboratory evaluation in which the aggregate gradation and the asphalt cement content were kept constant. The weight of the aggregate used to create each specimen was 1200 g to which 84 g (7%) of AC-20 asphalt cement was added for each specimen. Three different amounts of fibers were tested for each of the two types of nets obtained. The nylon trawl net was tested at 2.5% (2.1 g), 5.0% (4.2 g), and 10.0% (8.4 g) by weight of the asphalt used. The polypropylene gill net was tested at 5.0% (4.2 g), 10.0% (8.4 g), and 15.0% (12.6 g) by weight of the asphalt used. For each of these variations three specimens were prepared and tested. In addition to the fiber reinforced specimens, four control specimens containing no fiber were also prepared and tested.

It is also important to note that the problem of fiber clumping or balling discussed earlier did occur at times. This was more of a problem with the higher fiber percentages, and more noticeable with the nylon trawl netting than with the polypropylene gill net. The nylon fibers had a tendency to clump together even before the asphalt cement was added. It is for this reason that the amounts of nylon fibers used in this experiment were lower than those used for the polypropylene fibers. This problem could be reduced in the future by altering the shredding process of the nets. If the nets had been shredded to a lesser degree they would be more intact and less likely to clump. If they were shredded to a higher degree there would be a higher percentage of single fibers which would be easier to disperse into the mixture.

During the Marshall procedure each compacted test specimen is subjected to the following tests and analysis: (1) the bulk specific gravity determination ; (2) density-voids analysis; and (3) the stability and flow tests. This section will present and discuss the results of these tests and analysis for each of the different specimens prepared in the study.

## 3.2 BULK SPECIFIC GRAVITY

Before performing the density-voids analysis the bulk specific gravity of the specimen must be determined. The bulk specific gravity is obtained by dividing the specimen's weight in air by its volume. The average figure is then used to obtain the unit weight, or density, in pounds per cubic foot. The theoretical specific gravity is also calculated using an equation relating the percent asphalt used and the specific gravity of the asphalt and the aggregate (see appendix 6.2 for equation). The results are presented below in TABLE 2:

**TABLE 2**

**DENSITY & BULK SPECIFIC GRAVITY**

| SAMPLE TYPE | BULK SPECIFIC GRAVITY | THEORETICAL BULK S. G. | DENSITY (pcf) |
|---|---|---|---|
| CONTROL | 2.497 | 2.517 | 155.40 |
| 2.5 % NYLON | 2.478 | 2.517 | 154.60 |
| 5 % NYLON | 2.449 | 2.517 | 152.79 |
| 10 % NYLON | 2.405 | 2.518 | 150.09 |
| 5 % POLYPROP. | 2.473 | 2.517 | 154.32 |
| 10 % POLYPROP. | 2.455 | 2.518 | 153.17 |
| 15 % POLYPROP. | 2.420 | 2.519 | 151.04 |

The magnitude of these values achieved during compaction in the laboratory is not of great importance. The main purpose of calculating these values is for their use in the density-voids analysis. It should be noted that the numbers vary very slightly, decreasing with increasing fiber content. The maximum decrease in specific gravity and density observed was only 3.4 %.

### 3.3.0 DENSITY-VOIDS ANALYSIS

Two properties of the compacted specimen are determined in the density-voids analysis: the percent voids in the mineral aggregate and the air voids percent in the total mix. The air voids percentage represents the value obtained by dividing the bulk specific gravity of the specimen by the theoretical specific gravity, multiplying by 100 and then subtracting from 100 (see appendix 6.2 for equation)[8].

The air void % in a compacted specimen is suggested by most agencies to be between 3 and 5 % . Low air void % is important in order to minimize the aging of the asphalt cement films within the aggregate mass and also minimize the possibility that water can get into the mix, penetrate the thin asphalt cement film, and strip the asphalt cement off the aggregates[8]. As shown by the bar graph (FIGURE 1) the air void % of the specimens prepared are close to the specified range. For the most part the values seem slightly low. This could be caused by the fact that the amount of asphalt cement absorbed by the fibers and the aggregate could not be taken into account.

In general the air void % increased with increasing fiber content for both the nylon and polypropylene fibers. The values are presented in TABLE 3 at the end of this section. In both cases the air voids % of specimens with fibers is substantially higher than the control value. It is also important to note that the air void % for the nylon reinforced specimens were about one and a half times greater than the air void % for the polypropylene reinforced specimens. This could be due to the tendency of the nylon fibers to clump together and the lower density of the nylon fibers. Overall these results were as expected. The values are close enough to the suggested range to be acceptable and further optimization of the mix design could easily bring the value within the specified limits.

The percent voids in the mineral aggregate (VMA) represents that volume of the compacted specimen which is not occupied by aggregate and absorbed asphalt. This value is calculated using an equation which relates the bulk specific gravity of the specimen, the specific gravity of the aggregate, and the percent of asphalt cement used (see appendix

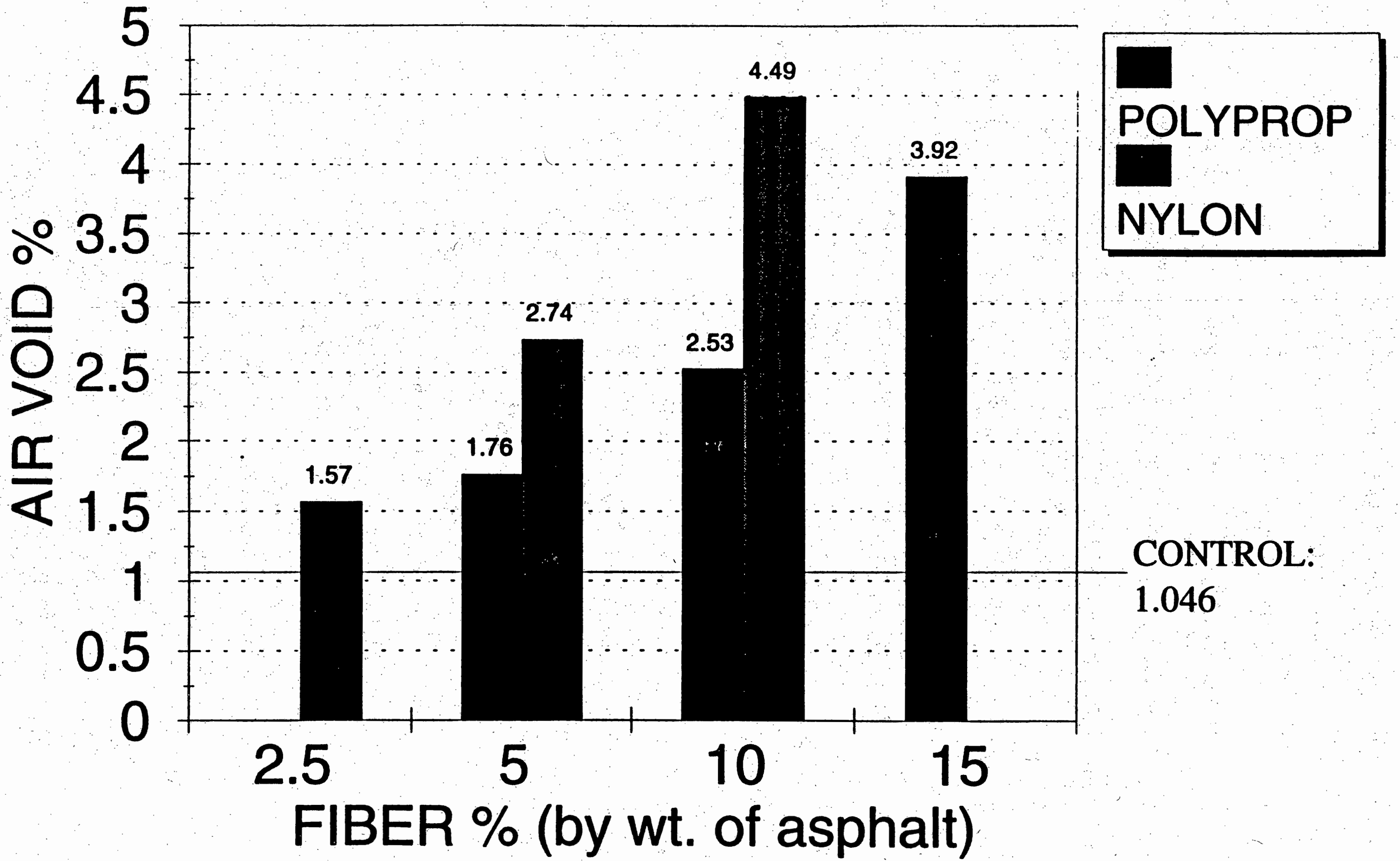


FIGURE 1 : AIR VOID % vs FIBER %

6.2 for equation). This total amount of voids significantly affects the performance of a mixture because if the VMA is to small, the mix may suffer durability problems, and if the VMA is to large, the mix may show stability problems and be economical to produce. The VMA should be sufficient to allow for thermal expansion of the asphalt and aggregate under summer temperatures without a loss in stability, yet low enough to keep out harmful air and moisture which could effect the durability of the pavement[8].

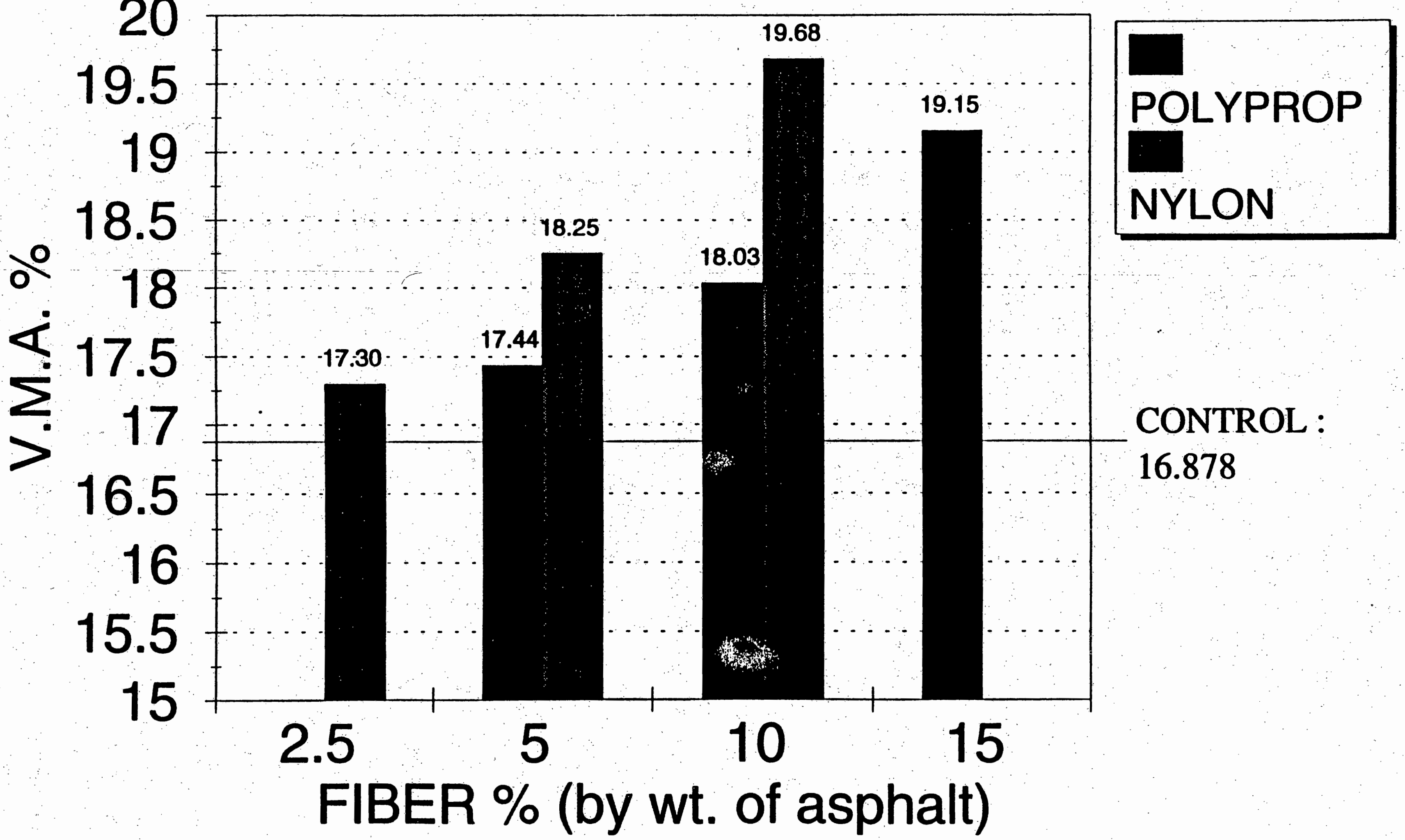
The recommended minimum VMA for the aggregate gradation used in this study has been proposed by the Asphalt Institute to be approximately 14% . As shown in the bar graph (FIGURE 2) and in TABLE 3 the values of VMA for all specimens prepared were above the minimum recommended VMA % and well within the normally acceptable range of VMA values. In general the VMA increased with increasing fiber content for both types of nets. In all cases the VMA was higher in the fiber reinforced specimens than in the control specimens. The VMA values were also higher for the specimens with the nylon netting compared with those containing the polypropylene fibers. Overall These values were very acceptable. In all cases the VMA was close enough to the recommended value that no adverse effects would be expected.

**TABLE 3**

**AIR VOID % & VMA %**

| SAMPLE | AIR VOID % | VMA % |
|---|---|---|
| CONTROL | 1.046 | 16.878 |
| 2.5 % NYLON | 1.570 | 17.296 |
| 5 % NYLON | 2.737 | 18.253 |
| 10 % NYLON | 4.491 | 19.683 |
| 5 % POLYPROP. | 1.764 | 17.436 |
| 10 % POLYPROP. | 2.531 | 18.035 |
| 15 % POLYPROP. | 3.916 | 19.155 |

## FIGURE 2 : VMA % vs FIBER %



### 3.4 STABILITY FLOW TEST

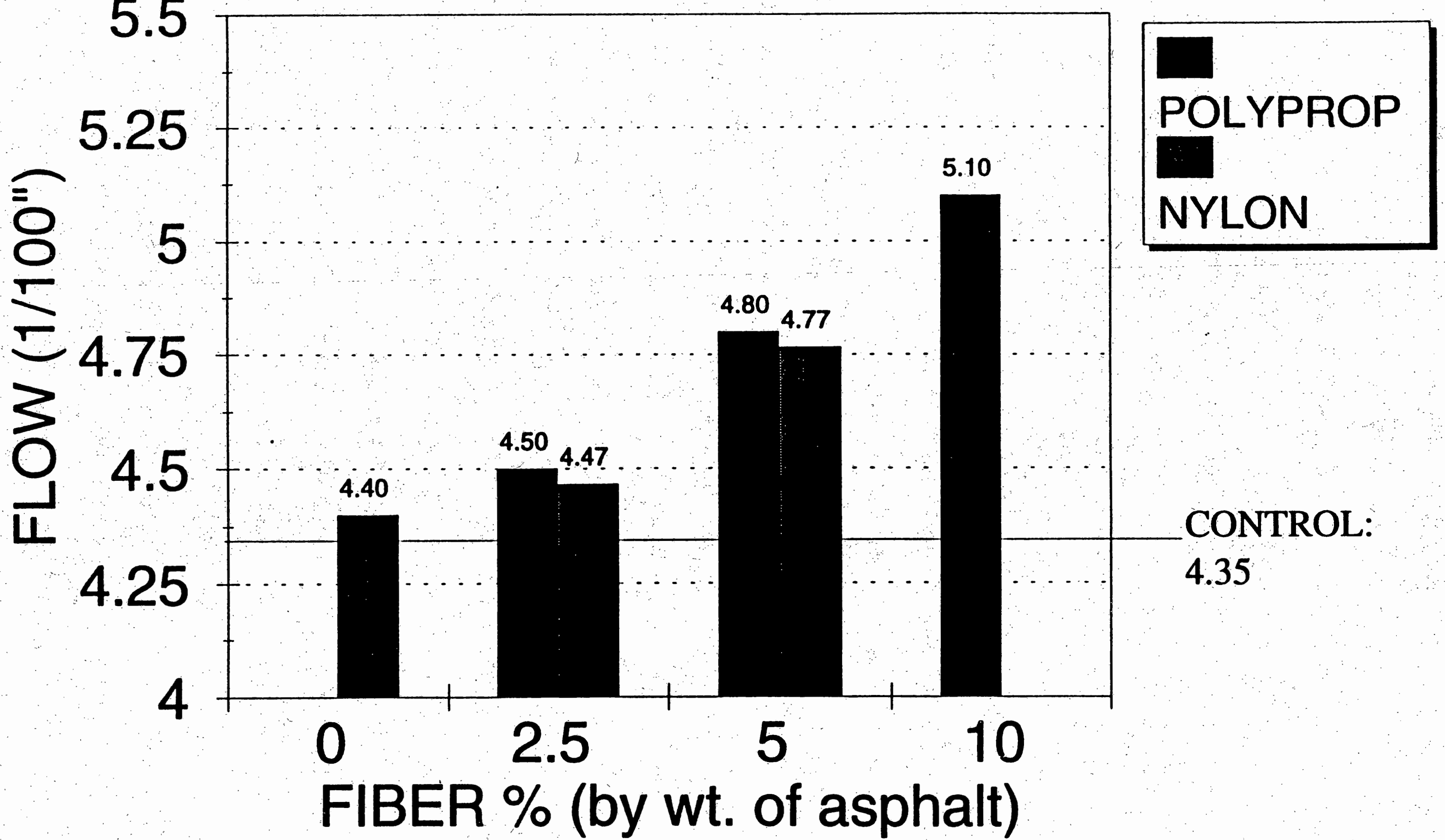
Two of the most important physical properties of the specimens are determined during the actual Marshall test: the flow and the stability. The flow value represents the total vertical deformation or strain occurring in the specimen between no load and maximum load during the stability test in hundredths of an inch. High flow values usually indicate a mix which will experience permanent deformation under traffic. Low flow values indicate a mix with higher than normal voids and insufficient asphalt for durability. Mixtures with low flow values may experience premature cracking due to mixture brittleness during the life of the pavement[8].

For Marshall designs subjected to 75-blows per side the maximum flow value is 16[8]. As shown in the bar graph (FIGURE 3) and summarized in TABLE 4, the values of flow for the specimens tested are well below this value. These values are also below the minimum recommended flow value of 8 . Since the samples do not contain higher than normal voids and have more than a sufficient amount of asphalt, it is expected that the flow meter was not working correctly. Although there is a trend in the results, all of the average values fell in the short range of 4 to 5 . As with all previous results the flow value increased with increasing fiber content for both types of fibers. It is also important to note that the flow values for both types of net were almost identical at the fiber contents of 5% and 10%.

Overall the flow values for all specimens were very low. This is more likely due to an error in the testing equipment than in the mix design. This must be studied much closer because if the values are accurate, the mix designs must be altered to increase the flow value. More importantly, it can be observed that the addition of fibers in the asphalt pavement did not have any significant adverse effects on the flow characteristics of the specimens.

The most important physical property measured in this study is the stability. The Marshall stability represents the maximum load resistance in pounds that a standardized

FIGURE 3 : FLOW vs FIBER %

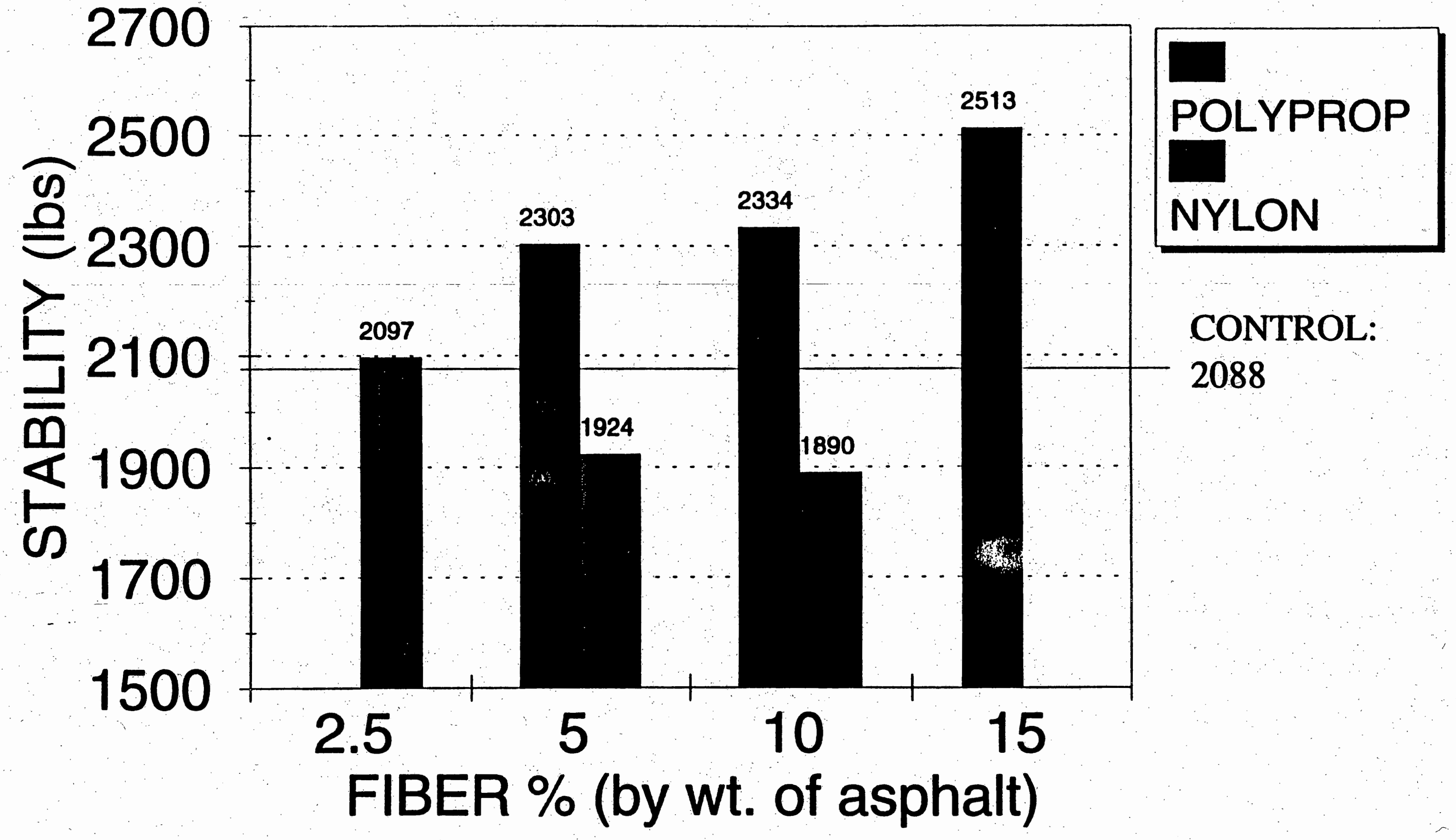


specimen will develop at 60°C at a loading rate of 2 inches per minute. The stability value is recorded by computer. Measured stability values for specimens which depart from the standard thickness of 2.5 inches are converted to an equivalent value by means of a conversion factor (see appendix 6.2 for calculations). The stability is the ability of the asphalt paving mixture to resist deformation from imposed loads[8].

Stability is dependent upon both internal friction an cohesion. The cohesion varies with the viscosity and amount of asphalt cement in the mixture. The internal friction is a function of the frictional and interlocking resistance of the aggregate in the mixture. Frictional resistance varies with the surface roughness and angularity of aggregate and the area particle contact. It is therefore important to take a close look to see how the addition of fibers to the mix affects the stability values.

The results of the stability test are presented in the bar graph (FIGURE 4) and summarized in TABLE 4. The Marshall design criteria for stability for a specimen subjected to 75 compaction blows on each side is 1800 lbs. At or above this value the pavement is suitable for use in high traffic areas. For none of the specimens was the average stability below this value. The average corrected stability for the four control specimens was 2088 lbs. For the nylon fibers at 2.5% the stability was only slightly improved at 2097 lbs. As more nylon fibers were added the stability decreased slightly. At %5 the stability was 1924 lbs and at 10% was 1890 lbs. The results for the nylon fibers therefore only showed improvement for the smallest amount of fiber addition. This is probably due to the affect of clumping of the fibers, which much more of a problem with the nylon fibers. This problem can be alleviated by finding a better way to disperse the fibers homogeneously throughout the mixture. In addition the mix design could be optimized for each fiber percentage, allowing for the best possible stability. Taking into account these problems the results were still encouraging. Even at the highest fiber percentage, with an unoptimized mix design and a high degree of fiber clumping, the decrease in stability was only 9.5%

FIGURE 4 : STABILITY vs FIBER %



For the polypropylene fibers the results were much better. The stability increased above the control value with increasing fiber amounts. At 5% fibers the stability was 2303 lbs, an increase of over 10%. At 10% fiber content the stability was 2334 lbs, an increase of almost 12%. At 15% fibers the stability was improved to 2513 lbs, an impressive increase of over 20%. The problem of fiber clumping was much less of a problem for these fibers possibly leading to the improvements in stability. These improvements could be further enhanced by optimizing the asphalt content and aggregate type and gradation in order to take full advantage of the effects of the fibers.

## TABLE 4

## MARSHALL FLOW & STABILITY

| SAMPLE TYPE | FLOW (1/100") | STABILITY (lbs) |
|---|---|---|
| CONTROL | 4.35 | 2088 |
| 2.5 % NYLON | 4.40 | 2097 |
| 5 % NYLON | 4.47 | 1924 |
| 10 % NYLON | 4.77 | 1890 |
| 5 % POLYPROP. | 4.50 | 2303 |
| 10 % POLYPROP. | 4.80 | 2334 |
| 15 % POLYPROP. | 5.10 | 2513 |

## 4.0 CONCLUSIONS & RECOMMENDATIONS

Overall the effects of the fibers on the properties of the specimens were acceptable. For the density-voids analysis the fibers did not cause the values to exceed the suggested range of values. For the stability results the nylon fibers caused some mixing problems and led to a slight decrease in the stability for fiber amounts. The nylon fibers did not adversely affect the stability enough to disregard its use in the mix at high values. On the other hand, the polypropylene fibers were very successful at increasing the stability, and even larger amounts of fiber asphalt could be considered.

The results of this study are therefore very promising. The polypropylene fibers increased the stability significantly with no adverse affects on any of the other properties measured. While the nylon fibers, although decreasing the stability slightly, also had no significant adverse affects on any of the other properties. It can be concluded that the positives are significant enough to justify the use of waste fishing nets in asphalt pavement.

One reason that these preliminary results are impressive is that it should be possible to improve them significantly through further research. By performing an extensive Marshall mix design, the optimum asphalt cement content for each fiber type and each fiber amount could be determined. In addition the aggregate gradation, aggregate type, and size and state of the fibers could be altered so that the positive affects of the fibers on the internal friction of the mixture could be optimized. The viscosity grade of the asphalt could also be varied to determine its affects on the properties of a fiber reinforced asphalt pavement.

Another important variation possible in the design of a fiber reinforced asphalt pavement, as discussed earlier, is the process of adding the fibers to the mix. The point at which the fibers are added to the mixture could significantly change the properties of the pavement. This concept should be combined with lab and field research done on the use of fiber reinforced asphalt pavements. Studies have been and are currently being conducted involving the improvement of the of the fiber addition process. Previous with fiber reinforced pavements has been plagued by inherent mix problems that could raise production costs and inhibit usage. Therefore the advantages and disadvantages of adding the fibers to either the aggregate (dry process) or the asphalt cement (wet process) on a large scale must be determined. Most of the successful work in this area involves a separate feed to input the fibers simultaneously with the aggregate. Experimentally, this is best represented by the dry process used in this study. The benefits, if any, of the wet process should be determined so that the results may be compared.

In addition to extensive variations in the mix design, it would be advantageous to study a wider range of properties of the specimens produced. Tests such as freeze-thaw resistance, tensile strength testing, penetration testing, and creep testing (as a measure of rut resistance), which were not available at this stage, should be performed. This will allow for a greater understanding of the affects o fishing net fibers in asphalt pavements.

In conclusion, there is little debate that a need exists to effectively deal with the problem of fishing net waste. With the recent push to incorporate waste materials in hot mix asphalt, a possible solution has become apparent. An important consideration is the affect of incorporating such a waste material on the quality and performance of the hat mix asphalt pavement. Of course it would not be economically feasible to complicate the production of asphalt pavements if the result was a pavement with a shorter life and higher maintenance costs. The results of this study show that the addition of fishing net waste fibers does not have any significant adverse affects on the properties of asphalt pavement specimens, and in many cases the fibers actually improve important properties. It is therefore imperative that more extensive research be undertaken so that fibers derived from fishing nets can begin to be incorporated into actual asphalt pavements.

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# 6.0 APPENDIX 1

## RAW DATA

## HOT MIX DESIGN DATA BY THE MARSHALL METHOD

**NYLON FIBERS:**

| Fiber % | Fiber | Asphalt | % | Corr. | Weight of Sample (grams) | | | Spec. Gravity | | Unit Wt. | Air Void | V.M.A. | Stability (lbs) | | Flow |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| by wt. of AC | (grams) | (grams) | AC | Ratio | Dry | W/Wax | Volume | Bulk | Theor. | (pcf) | % | % | Meas. | Adj | (1/100") |
| 2.5-A | 2.10 | 84.00 | 6.53 | 1.04 | 1237.30 | 1242.40 | 506.50 | 2.468 | 2.517 | 153.98 | 1.96 | 17.624 | 1991.00 | 2070.64 | 4.30 |
| 2.5-B | 2.10 | 84.00 | 6.53 | 1.04 | 1239.50 | 1245.00 | 504.80 | 2.482 | 2.517 | 154.91 | 1.37 | 17.131 | 1938.00 | 2015.52 | 4.50 |
| 2.5-C | 2.10 | 84.00 | 6.53 | 1.04 | 1221.10 | 1226.20 | 497.00 | 2.482 | 2.517 | 154.90 | 1.38 | 17.133 | 2120.00 | 2204.80 | 4.40 |
| **AVERAGE:** | | | | | | | | **2.478** | | **154.598** | **1.570** | **17.296** | | **2096.99** | **4.400** |
| 5.0-A | 4.20 | 84.00 | 6.52 | 1.00 | 1245.70 | 1252.40 | 515.50 | 2.448 | 2.517 | 152.77 | 2.75 | 18.262 | 2167.00 | 2167.00 | 4.50 |
| 5.0-B | 4.20 | 84.00 | 6.52 | 1.04 | 1205.80 | 1210.50 | 497.50 | 2.447 | 2.517 | 152.68 | 2.81 | 18.311 | 1675.00 | 1742.00 | 4.30 |
| 5.0-C | 4.20 | 84.00 | 6.52 | 1.04 | 1200.30 | 1207.50 | 497.00 | 2.451 | 2.517 | 152.92 | 2.66 | 18.186 | 1792.00 | 1863.68 | 4.60 |
| **AVERAGE:** | | | | | | | | **2.449** | | **152.791** | **2.737** | **18.253** | | **1924.23** | **4.467** |
| 10.0-A | 8.40 | 84.00 | 6.50 | 0.96 | 1226.70 | 1233.70 | 525.00 | 2.368 | 2.518 | 147.77 | 5.96 | 20.920 | 2130.00 | 2044.80 | 4.90 |
| 10.0-B | 8.40 | 84.00 | 6.50 | 0.96 | 1250.90 | 1258.70 | 525.80 | 2.415 | 2.518 | 150.69 | 4.11 | 19.360 | 1849.00 | 1775.04 | 4.60 |
| 10.0-C | 8.40 | 84.00 | 6.50 | 1.00 | 1247.20 | 1254.50 | 520.00 | 2.433 | 2.518 | 151.79 | 3.40 | 18.768 | 1850.00 | 1850.00 | 4.80 |
| **AVERAGE:** | | | | | | | | **2.405** | | **150.085** | **4.491** | **19.683** | | **1889.95** | **4.767** |

| | |
|---|---|
| Gac: | 1.029 |
| Geff: | 2.80 |

## HOT MIX DESIGN DATA BY THE MARSHALL METHOD

**POLYPROPYLENE FIBERS:**

| Fiber % | Fiber | Asphalt | % | Corr. | Weight of Sample (grams) | | | Spec. Gravity | | Unit Wt. | Air Void | V.M.A. | Stability (lbs) | | Flow |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| by wt. of AC | (grams) | (grams) | AC | Ratio | Dry | W/Wax | Volume | Bulk | Theor. | (pcf) | % | % | Meas. | Adj | (1/100") |
| 5.0-A | 4.20 | 84.00 | 6.52 | 1.04 | 1233.40 | 1239.80 | 506.00 | 2.469 | 2.517 | 154.05 | 1.93 | 17.579 | 2079.00 | 2162.16 | 4.20 |
| 5.0-B | 4.20 | 84.00 | 6.52 | 1.04 | 1234.70 | 1241.00 | 505.80 | 2.472 | 2.517 | 154.24 | 1.81 | 17.475 | 2134.00 | 2219.36 | 4.40 |
| 5.0-C | 4.20 | 84.00 | 6.52 | 1.04 | 1229.60 | 1236.00 | 502.50 | 2.479 | 2.517 | 154.66 | 1.55 | 17.253 | 2430.00 | 2527.20 | 4.90 |
| **AVERAGE:** | | | | | | | | **2.473** | | **154.319** | **1.764** | **17.436** | | **2302.91** | **4.500** |
| 10.0-A | 8.40 | 84.00 | 6.50 | 1.04 | 1221.90 | 1228.10 | 505.20 | 2.449 | 2.518 | 152.80 | 2.76 | 18.231 | 2152.00 | 2238.08 | 4.70 |
| 10.0-B | 8.40 | 84.00 | 6.50 | 1.04 | 1226.30 | 1233.10 | 506.10 | 2.456 | 2.518 | 153.26 | 2.47 | 17.985 | 2335.00 | 2428.40 | 4.50 |
| 10.0-C | 8.40 | 84.00 | 6.50 | 1.04 | 1228.50 | 1234.20 | 505.30 | 2.459 | 2.518 | 153.44 | 2.36 | 17.888 | 2245.00 | 2334.80 | 5.20 |
| **AVERAGE:** | | | | | | | | **2.455** | | **153.165** | **2.531** | **18.035** | | **2333.76** | **4.800** |
| 15.0-A | 12.60 | 84.00 | 6.48 | 1.09 | 1183.70 | 1189.10 | 490.50 | 2.440 | 2.519 | 152.26 | 3.14 | 18.499 | 2943.00 | 3207.87 | 5.20 |
| 15.0-B | 12.60 | 84.00 | 6.48 | 1.04 | 1196.80 | 1202.30 | 505.00 | 2.396 | 2.519 | 149.51 | 4.89 | 19.972 | 1873.00 | 1947.92 | 5.60 |
| 15.0-C | 12.60 | 84.00 | 6.48 | 1.00 | 1253.40 | 1258.20 | 521.60 | 2.425 | 2.519 | 151.34 | 3.72 | 18.993 | 2384.00 | 2384.00 | 4.50 |
| **AVERAGE:** | | | | | | | | **2.420** | | **151.038** | **3.916** | **19.155** | | **2513.26** | **5.100** |

| | |
|---|---|
| Gac: | 1.029 |
| Geff: | 2.80 |

# HOT MIX DESIGN DATA BY THE MARSHALL METHOD

**CONTROLS:**

| Sample | Asphalt | % | Corr. | Weight of Sample (grams) | | | Spec. Gravity | | Unit Wt. | Air Void | V.M.A. | Stability (lbs) | | Flow |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| No. | (grams) | AC | Ratio | Dry | W/Wax | Volume | Bulk | Theor. | (pcf) | % | % | Meas. | Adj | (1/100") |
| A | 84.00 | 6.54 | 1.14 | 1178.20 | 1183.90 | 477.60 | 2.497 | 2.517 | 155.80 | 0.79 | 16.665 | 1768.00 | 2015.52 | 4.00 |
| B | 84.00 | 6.54 | 1.05 | 1220.30 | 1226.20 | 495.80 | 2.491 | 2.517 | 155.43 | 1.02 | 16.859 | 2019.00 | 2119.95 | 4.50 |
| C | 84.00 | 6.54 | 1.03 | 1249.60 | 1254.90 | 508.30 | 2.484 | 2.517 | 155.02 | 1.29 | 17.080 | 1951.00 | 2009.53 | 4.80 |
| D | 84.00 | 6.54 | 1.04 | 1204.60 | 1209.80 | 489.10 | 2.489 | 2.517 | 155.34 | 1.08 | 16.911 | 2121.00 | 2205.84 | 4.10 |
| AVERAGE: | | | | | | | 2.490 | | 155.396 | 1.046 | 16.878 | | 2087.71 | 4.350 |

| | |
|---|---|
| Gac: | 1.029 |
| Geff: | 2.80 |

## 7.0 APPENDIX 2 : EQUATIONS

**Nomenclature:**

%AC : percent of aspahlt by weight of mix
SG : specific gravity
$G_{eff}$ : specific gravity of aggregate
$G_{ac}$ : specific gravity of asphalt cement
VMA : voids in mineal aggregate

**Equations:**

$$(1)\quad \%AC = \frac{\text{wt. of asphalt}}{\text{wt. of mix (asphalt + aggregate + fibers)}}$$

$$(2)\quad \text{BULK SG} = \frac{\text{dry wt.}}{\text{volume - (wt. w/wax - dry wt.)}}$$

$$(3)\quad \text{THEOR SG} = \frac{100}{\frac{1 - (\%AC/100)}{G_{eff}} + \frac{(\%AC/100)}{G_{ac}}}$$

$$(4)\quad \text{AIR VOIDS \%} = 100 - \left\{100 \times \frac{\text{BULK SG}}{\text{THEOR SG}}\right\}$$

$$(5)\quad \text{VMA \%} = 100 \times \left\{1 - \frac{\text{BULK SG} \times (\%AC/100)}{G_{eff}}\right\}$$

$$(6)\quad \text{Adj. Stability} = \text{Stability} \times \text{Correction Factor}$$

# RESULTS SET IV

# TEST PROGRAM ON HOT ASPHALT MIXTURE WITH FIBERS USING SUPERPAVE SCHEDULE

Department of Civil & Environmental Engineering
New Jersey Institute of Technology

November 10, 1997

INTRODUCTION

IMPORTANCE OF USING SUPERPAVE LEVEL 3

STUDY SCHEME

EXPERIMENT WORK

# INTRODUCTION

The research program of reinforcing the asphalt concrete pavement with plastic fibers consist of two parts of test process.

PART 1. Marshall Test:

A traditional empirical test method for HAM design, being performed at NJIT.

PART 2. SUPERPAVE LEVEL 3:

It is a test method to determine some fundamental properties using specimens that have undergone specific preconditioning.

Two new, sophisticated pieces of laboratory equipment-Repetitive Simple Shear Tester (RSST) and Indirect Tension Tester (IDTT) will be used to measure specific engineering properties of the laboratory compacted asphalt mix.

Performance is predicted on the basis of statistical correlation between laboratory results obtained under a given set of conditions and field observations.

Asphalt content can be selected based on desired pavement performance in fatigue and permanent deformation.

This is achievable with the current state of knowledge and is basically the approach proposed by some other researches.

It is the main part of the whole experiment program.

# IMPORTANCE OF USING SUPERPAVE LEVEL 3

Comparing with other test methods, the importance to use Superpave Level 3 is obvious.

From Marshall Test, values of Marshall stability and flow are recorded. Since it is an empirical test method, it is difficult to establish the relationships between the records and some important fundamental properties, such as resistance to fatigue cracking, rutting and low-temperature cracking. Actually there are no such correlation available now.

For Superpave Level 1, specimens are fabricated under a given set of conditions to determine their volumetric characteristics. Asphalt content is based on compactability data. There are no methods to predict the fundamental performance properties.

For Level 2, it is basically the concept followed by the Marshall test. Limits are set on those variables based on prior experience. Asphalt content is based on limits, ranges, or extreme values of the variables evaluated. Still fundamental performance properties are not available here.

For Level 4, it consists of the determination of fundamental properties of the mix (and /or components) and evolution of those properties with time, aging, strain and tress levels and moisture. Predication of behavior is made through an elaborate set of computer simulations. This approach is beyond the current state of knowledge. Asphalt content would be selected based on predicted pavement performance, which would be very close to actual performance.

In a word, Superpave Level 3 is the only method available to predict how well HAM will perform in the field based on some fundamental properties.

## STUDY SCHEME

1. A typical phase of pavement section would be selected to design for this study. Design requirement, supposed information of all the engineering conditions (traffic loading, geotechnical condition and climate data) would be determined with the help of NJDOT.

2. For comparison, both HAM with and without fibers will be designed in parallel for the different pavement section under the same design perquisite conditions.

3. RSST will be running to evaluate the rutting propensity of the mixes, such as the No. of cycles that yields maximum allowable shear strain under the preconditioning requirement of design.

4. IDTT will be performed to get the important mechanical engineering parameters, such as time & temperature dependent stiffness and failure limits at low in-service temperatures.

5. Analysis the test results of RSST to predict fatigue resistance property for each pavement.

6. Applying the results from IDTT to the strength analysis of the pavement sections, to see if the low temperature failure would be happened.

7. Conclusion.

## EXPERIMENT WORK

### Selection of Nylon Fibers:

The ultimate objective of the research is to specify using recycled plastic fibers for HAM blending. In this phase, we are going to try nylon fibers and start with virgin fibers. Two kind of monofil nylon fiber would be used for choice, 20 dpf and 45 dpf , which are supplied by Dupont.

### Specimen Variable Factors:

The factors on fiber's length, diameter, blending stiffness, curling shape influence, physical and chemical surface treatment would be investigated in the study.

Preliminary test on bonding properties between fiber and binder/HAM would be carried on to determine the selection of the length of fiber.

Procedure to Evaluate the Rutting Propensity of a Mix:

(1). Determine the number of ESALs for the design life;
(2). Select the maximum allowable rut depth;
(3). Determine mean highest 7-day pavement temperature on site at a depth of top layer.
(4). Execute RSST at constant height (CH) at the ESALs and the temperature;
(5). Determine the number of cycles in RSST that yields maximum allowable shear strain, giving the relationship between shear strain and the number of cycles obtained from RSST.

Scope of Indirect Tension Test:

(1). Determine temperature dependent elastic modules suitable for linear elastic layer analysis of the asphalt concrete layer;

(2). Determine time and temperature dependent stiffness suitable for linear viscoelastic or pseudo-viscoelastic analysis of thermal response of the asphalt concrete layer.

(3). Determine time and temperature dependent failure limits at low in-service temperatures(stress, strain, energy).

Other Preparations:

Since the testers are not established in NJIT, a test center must be chosen for the study program. Work shop training for using these testers are needed.