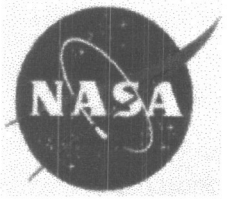

Characterization of M40J Desized and Finished Fibers

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Albuquerque, NM
and
E. Eugene Shin, Linda McCorkle,
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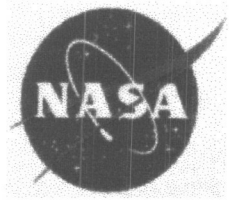
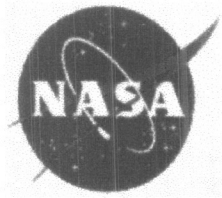
Presentation Outline

- Program Goals and Prior Year Results Summary
- Continuous Desizing and Finishing System Development
- Characterization of Desized and Finished M40J Carbon Fibers
- Conclusions and Future Work



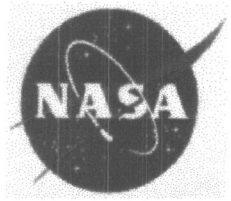
Problem Statement

- Toray M40J carbon fibers have an unusual combination of stiffness and strength for use in high-temperature structural applications
- Epoxy sizes on these fibers are not optimized for compatibility with high-temperature polymers



FY 00 Technical Approach

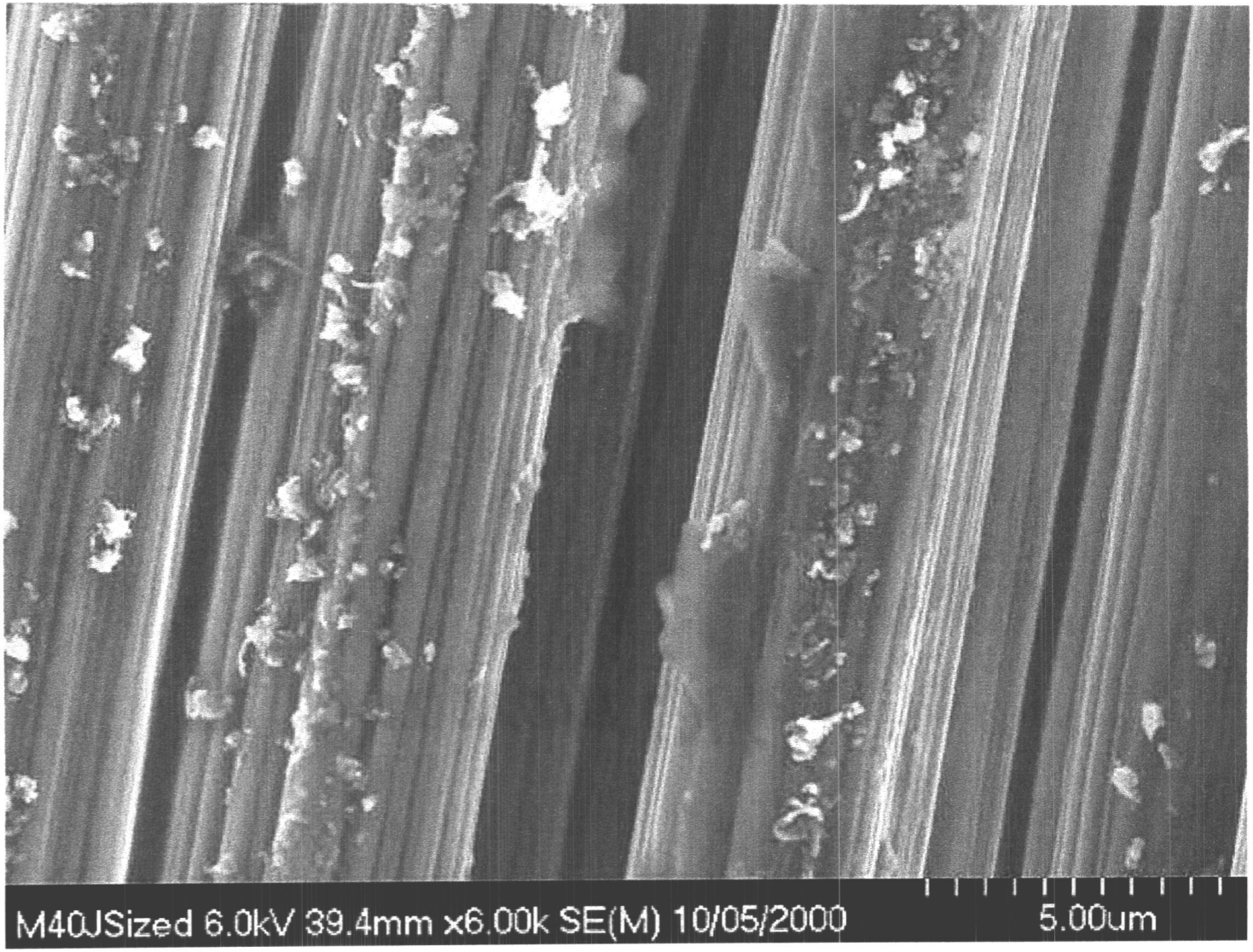
- Compare sized and chloroform desized (5 min.) fibers:
  - surface chemistry by XPS
  - surface energy by wetting
  - topography by SEM
- Characterize PMR-II-50 resin:
  - surface energy by wetting
  - surface chemistry by XPS



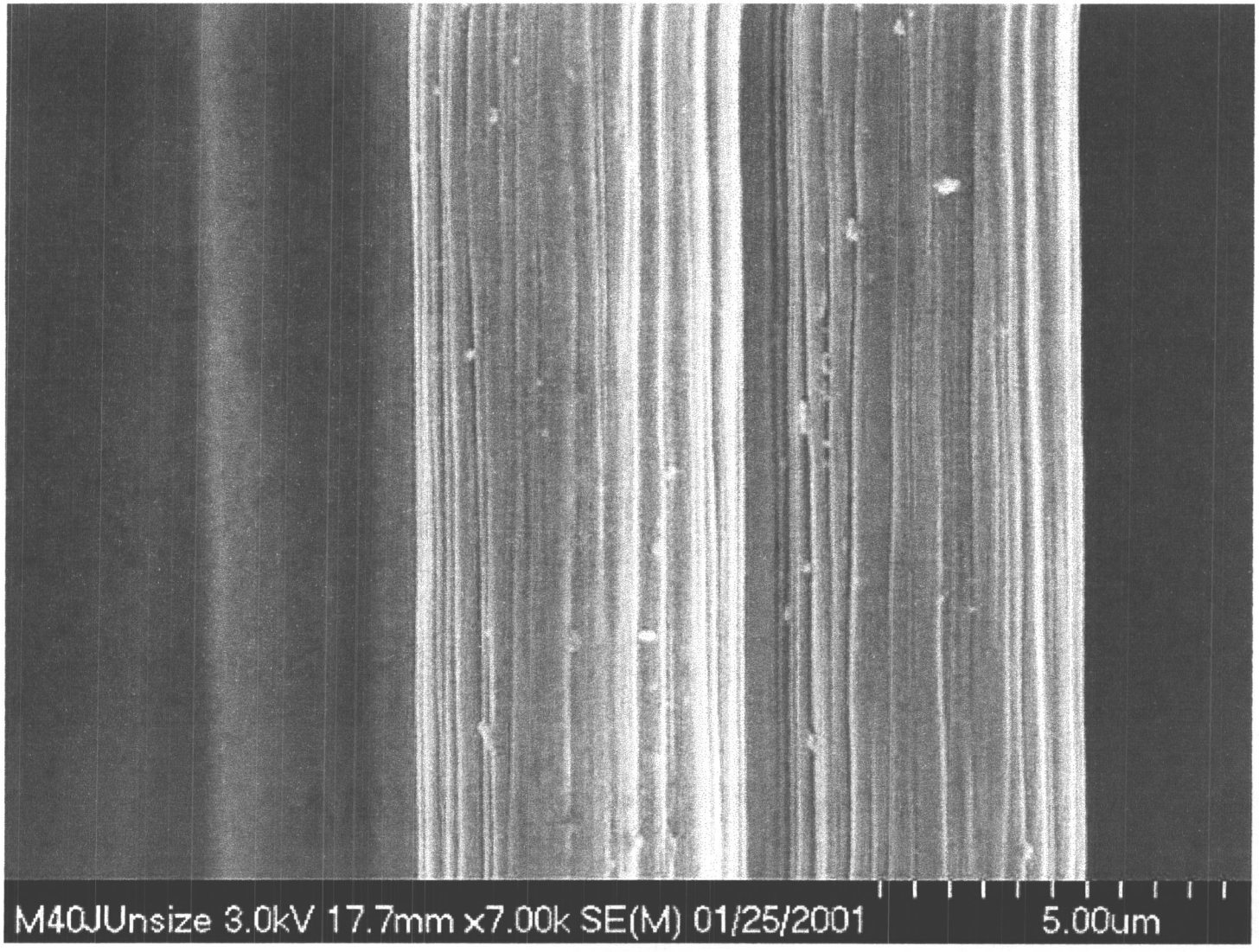
Program Objective

To search for empirical correlations between interfacial measurements and composite performance in order to develop effective high-temperature surface treatments.

Toray Sized M40J



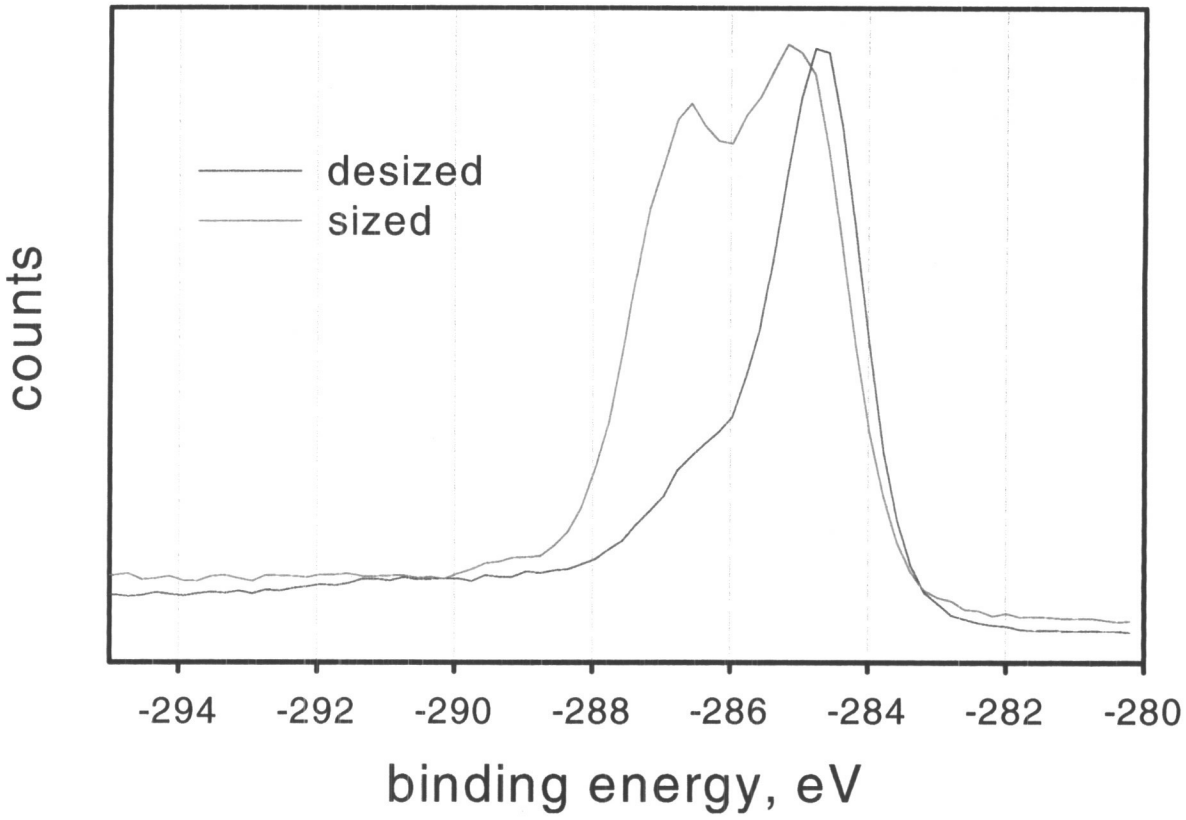
Toray M40J Desized



Wetting Results

1. Sized and desized surfaces are energetically and topographically heterogeneous
2. Formamide wetting shows that M40J surfaces and M60J surfaces display similar acidity whether sized or desized.
3. Ethylene glycol wetting suggests that size covers basic functionality that is uncovered by desizing
4. PMR-II-50 resin is mildly amphoteric, since W^{a-b} is small but finite for both acid-base probe liquids

XPS Analysis



Toray M40J carbon fiber

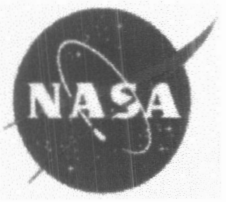
XPS Elemental Analysis - M40J Carbon Fibers

	Sized	Desized
%C-C	39.8	70.4
%C-O	34.4	13.3
%C-OO	2.1	0.9
%O1s	23.7	15.3

FY 00 Conclusions

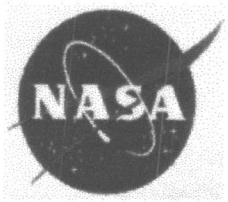
- Toray size coverage is very nonuniform
- Size contains predominantly hydroxyl groups, and shows slightly acidic character
- Desizing in hot chloroform leaves mostly clean fiber with small nodules of residual size
- Desized fiber surface is amphoteric with 12-15% oxygen moieties

FY 00 Conclusions (concluded)



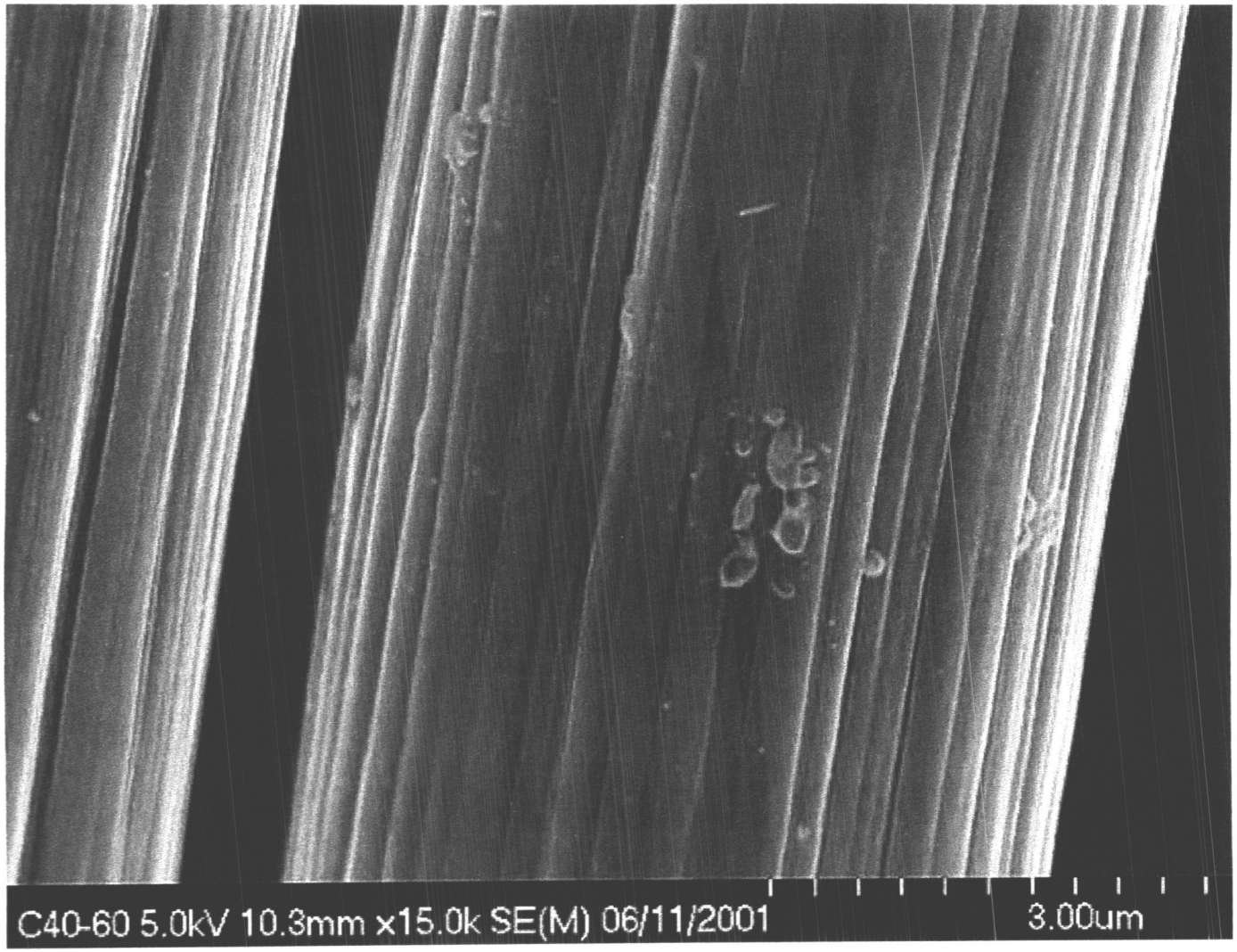
- PMR-II-50 resin also amphoteric
- Toray fibers highly striated
- High-temperature applications will require better sizing removal and/or surface treatment to remove residual size
- Previous work on unsized fiber indicates that the desized Toray fiber surface should be receptive to surface treatments and finishes

FY 01 Program Goals

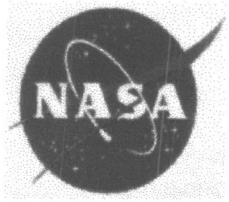


- Determine Operating Parameters for Continuous Desizing Line on M40J
- Fabricate continuous desizing unit with in-line finishing capability
  - Characterize resultant fibers
  - Produce large batches of desized and finished fibers for composite fabrication
- Fabricate and test unicomposites for interfacial adhesion

M40J Carbon Fiber Residual Size after 60 sec Chloroform Soak

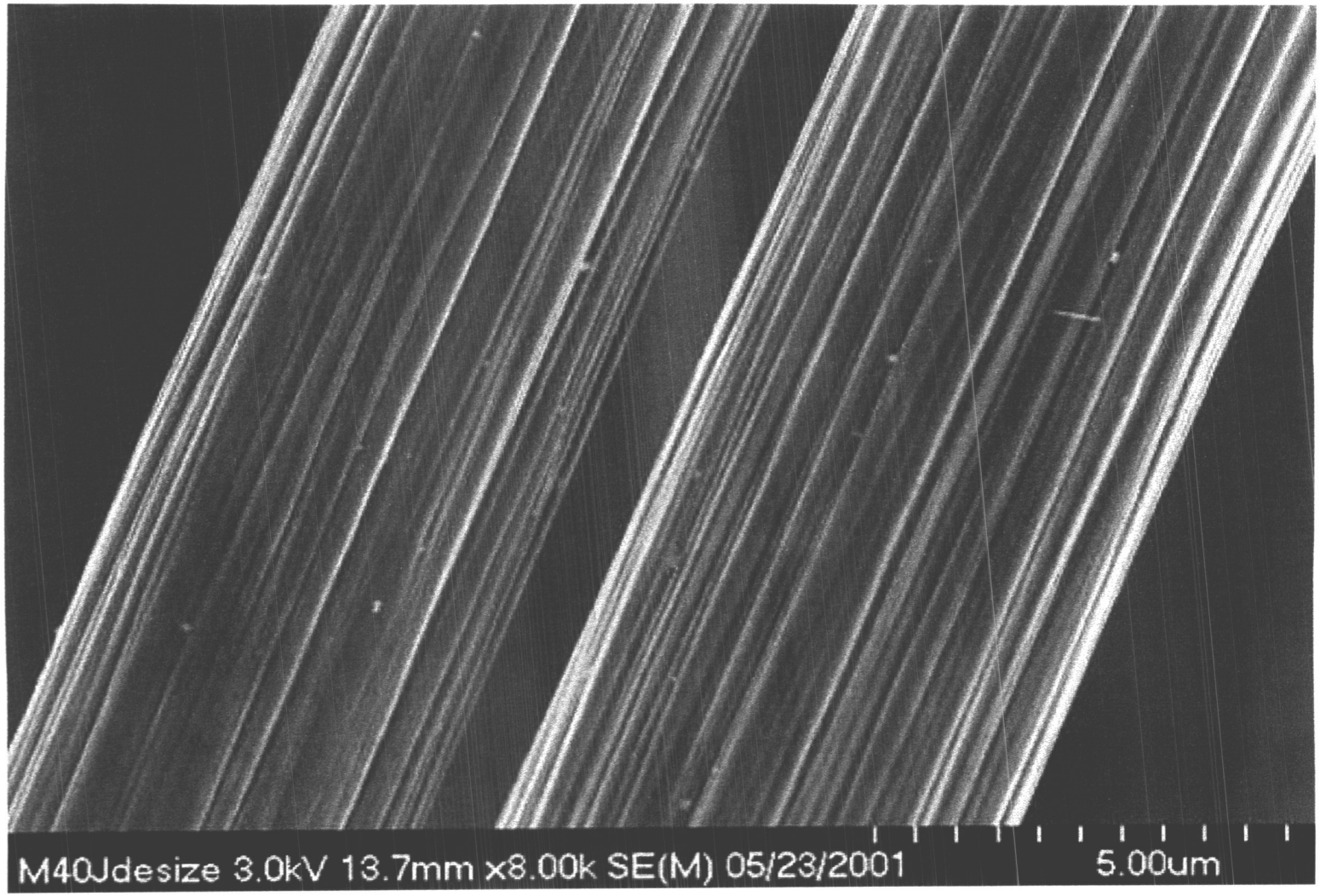


Desizing/Finishing System

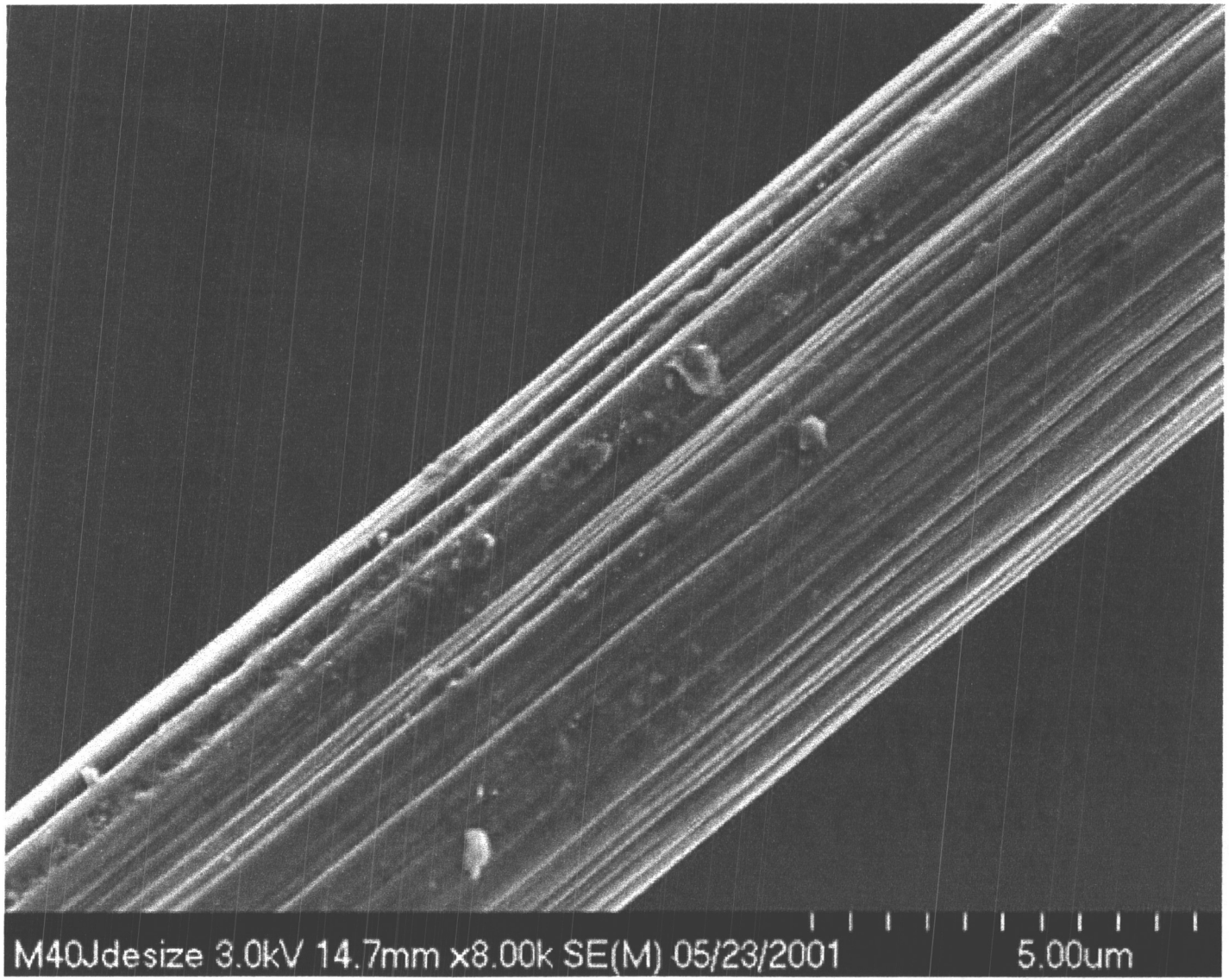


- 7 meter long 2 cm dia steel tube in and out of tank containing 25 cm dia wheel
- filled with heated chloroform
- tension controlled feed and take up at 1.6 meters/minute
- in-line drying furnace and finishing bath
- added ultrasonic transducer to return tank

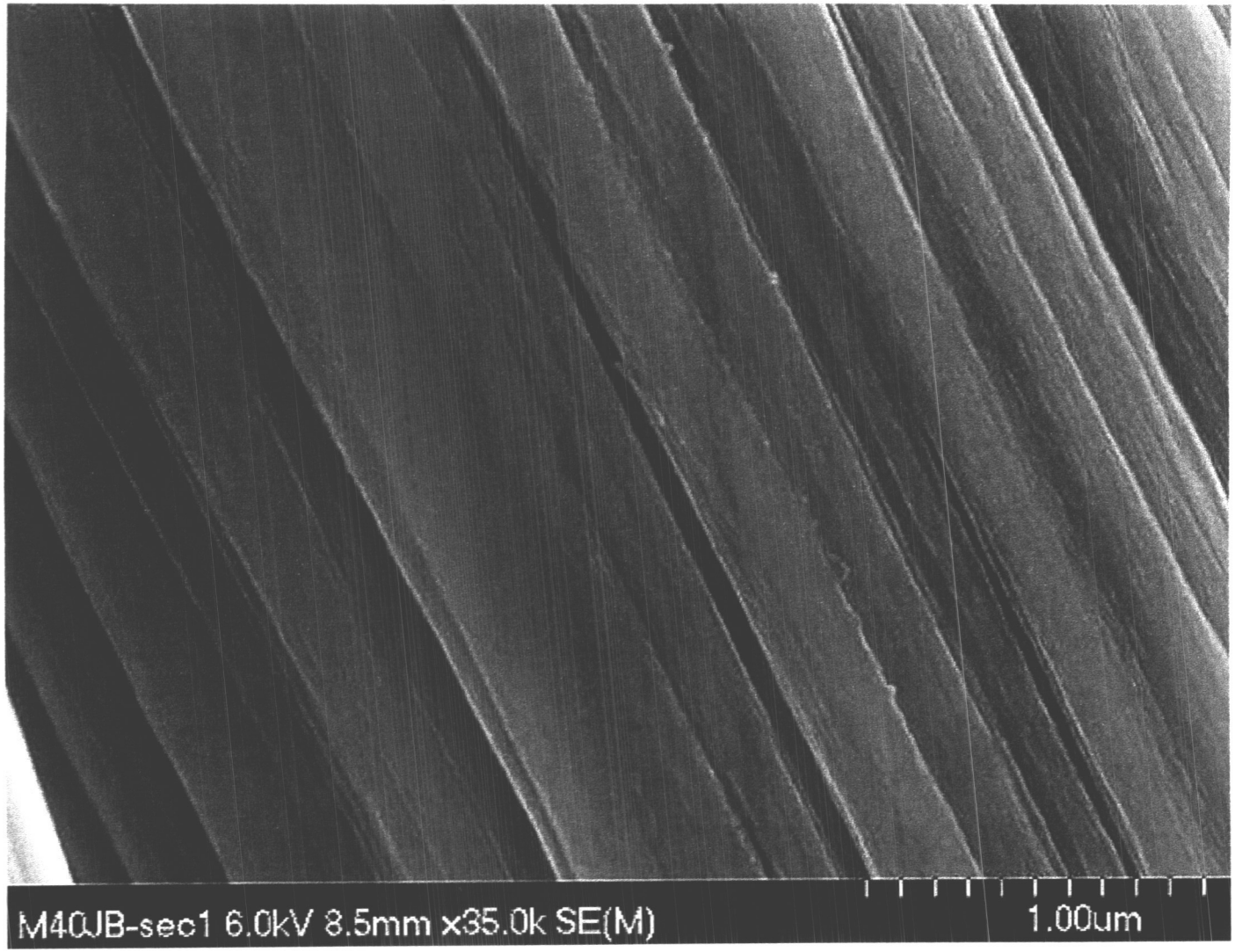
Appearance of Continuous Desized M40J Carbon Fibers



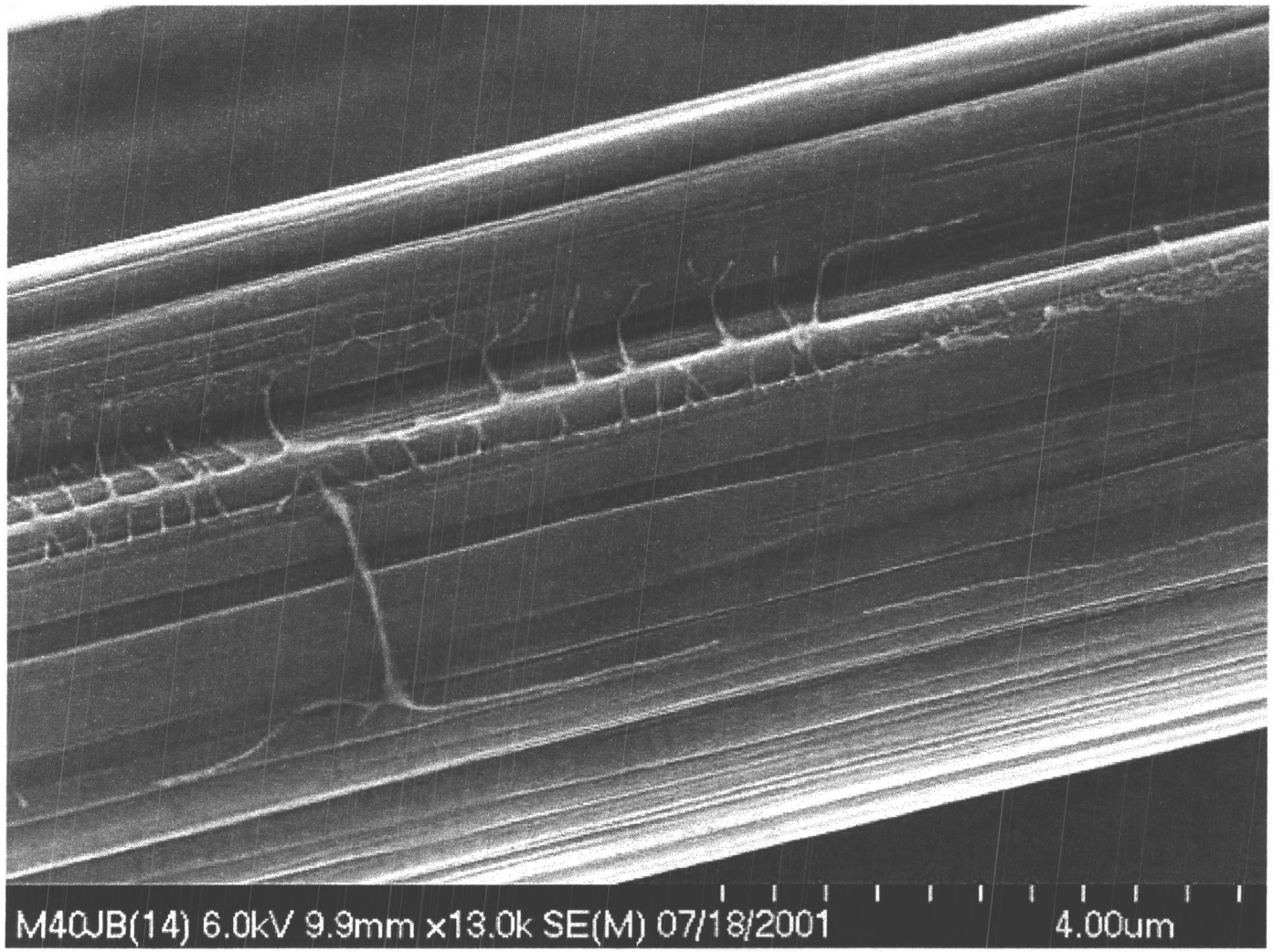
Appearance of Continuous Desized M40J Carbon Fibers



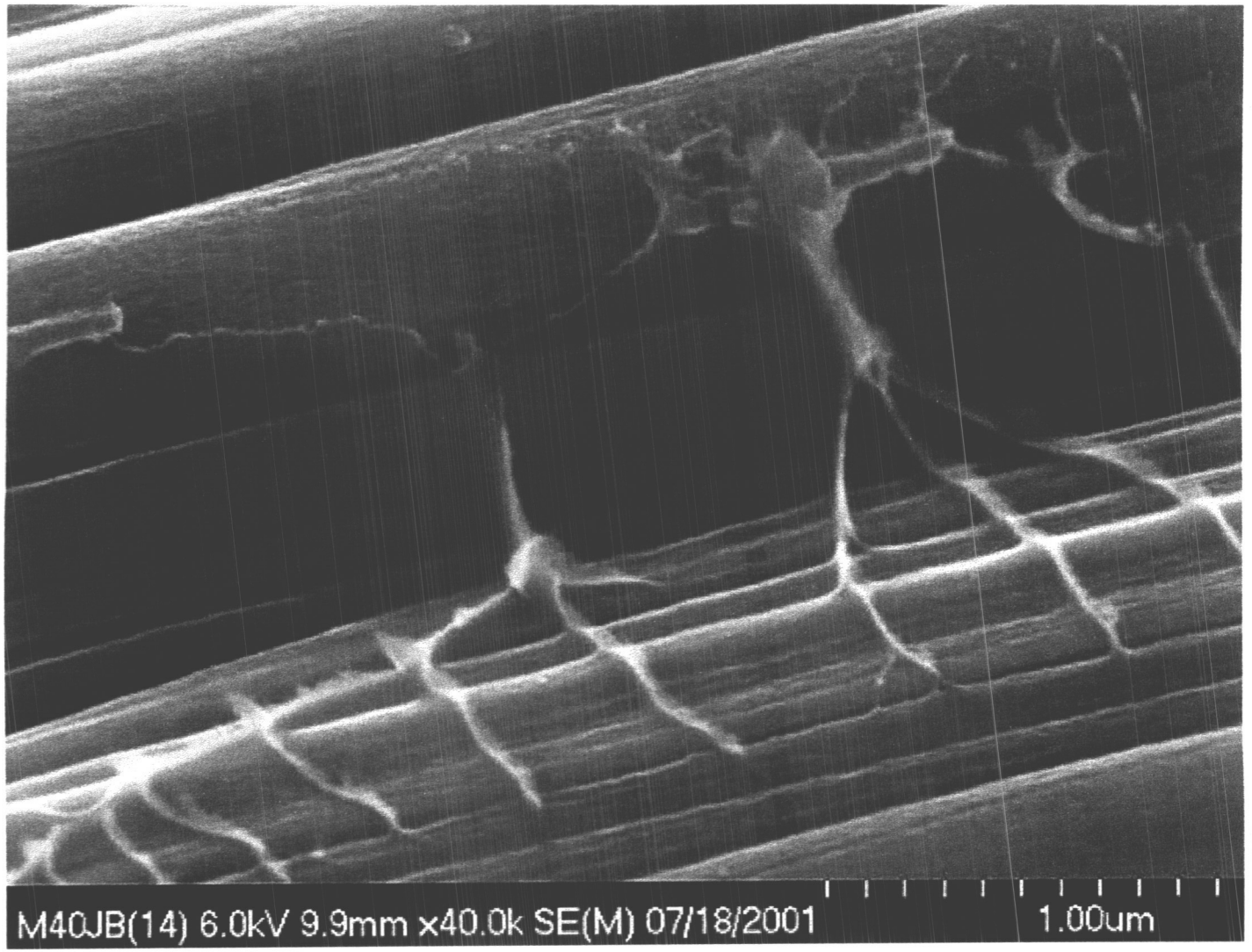
Appearance of M40J Carbon Continuously Desized w/Ultrasound



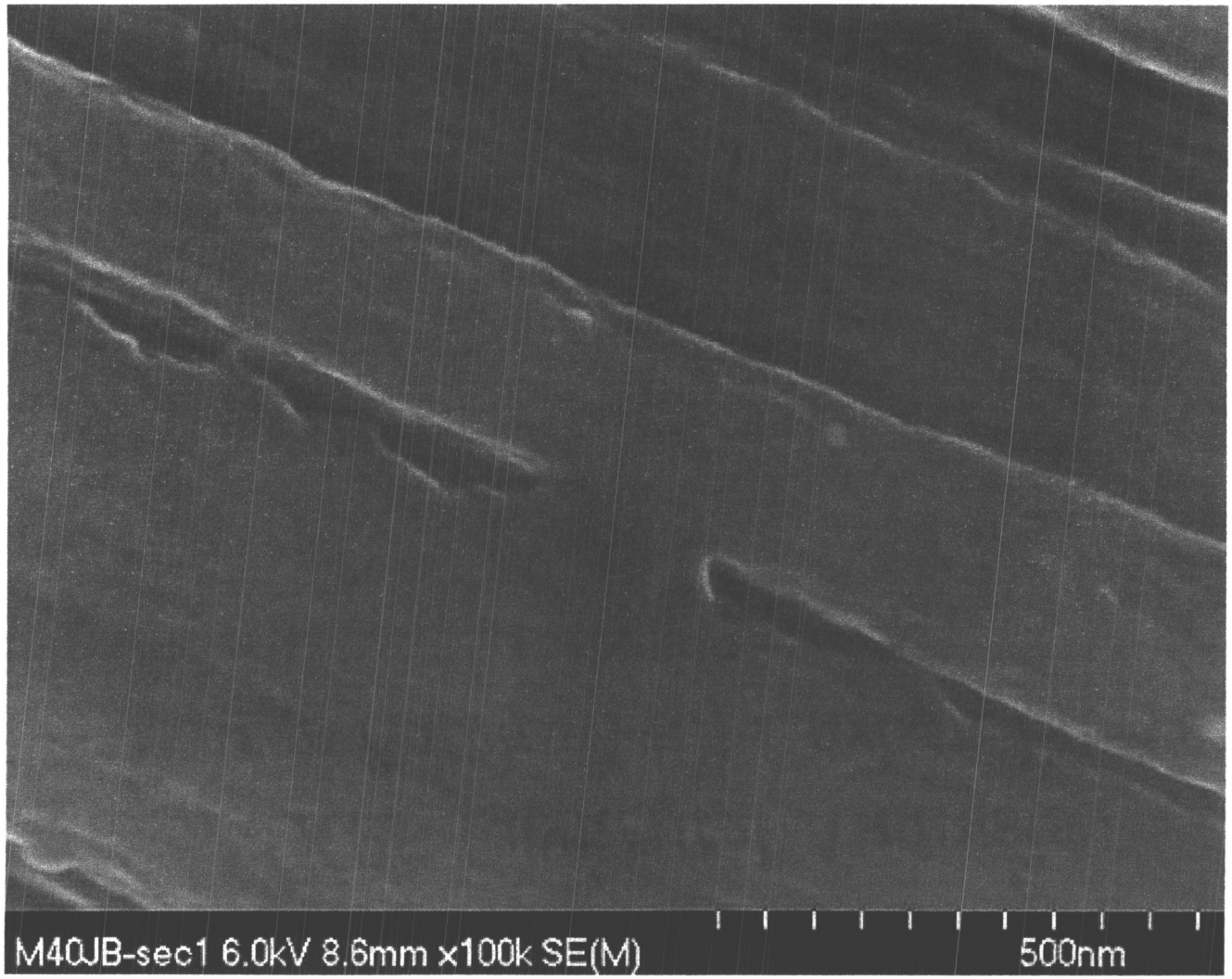
Desizing Intermediate Stage



High Magnification of Desizing Intermediate Stage



High Magnification Appearance of M40J Carbon Continuously Desized w/Ultrasound



XPS Elemental Analysis
Large Batch M40J Carbon Fibers

Section	%O	%C
1	9.6	90.4
1	11.9	88.2
2	14.1	85.9
2	13.0	87.0
3	11.2	88.8
3	10.5	89.5

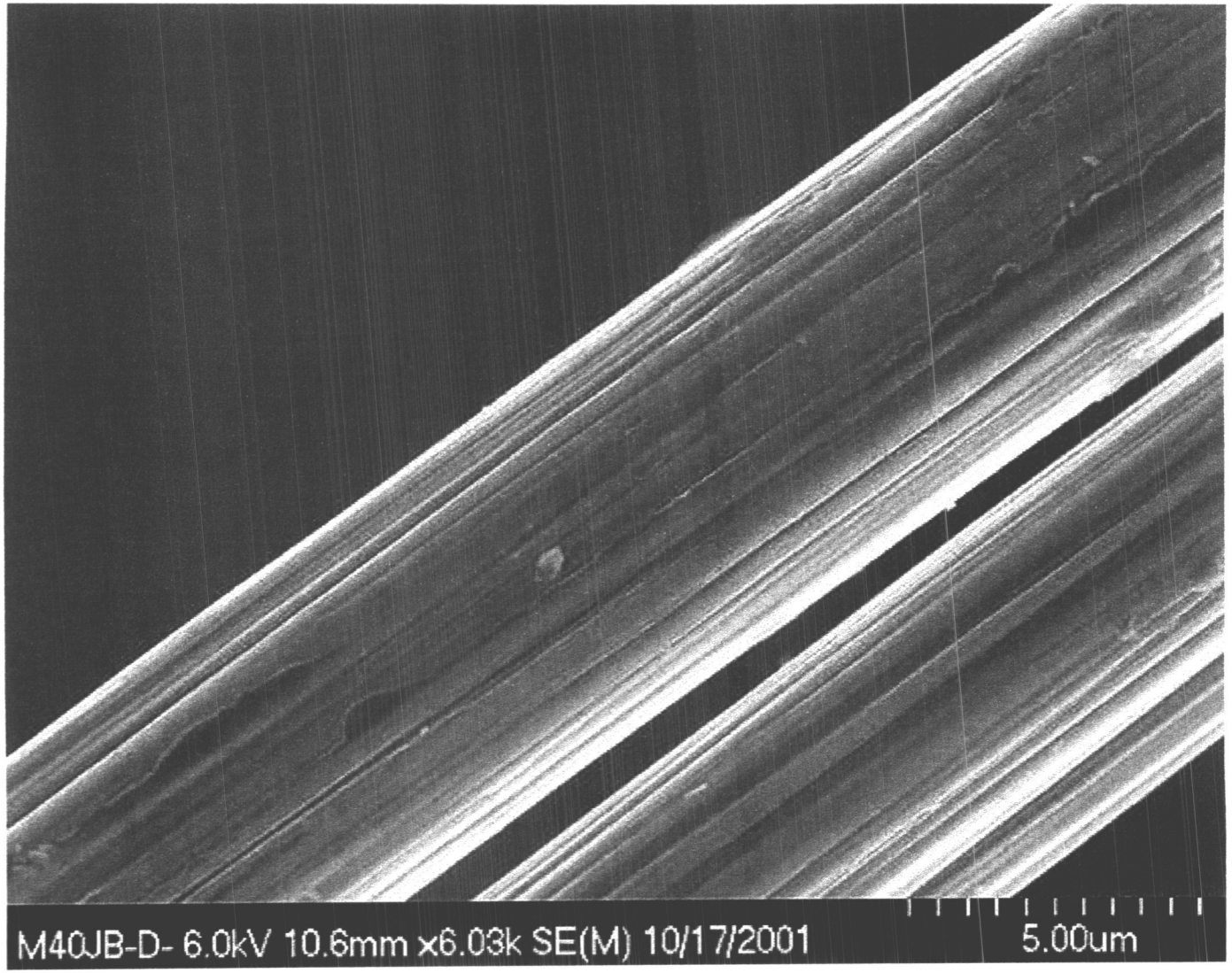
Reactive Finish Formulation

1. ATI 9307 Reactive Coupling Agent (0.3%)

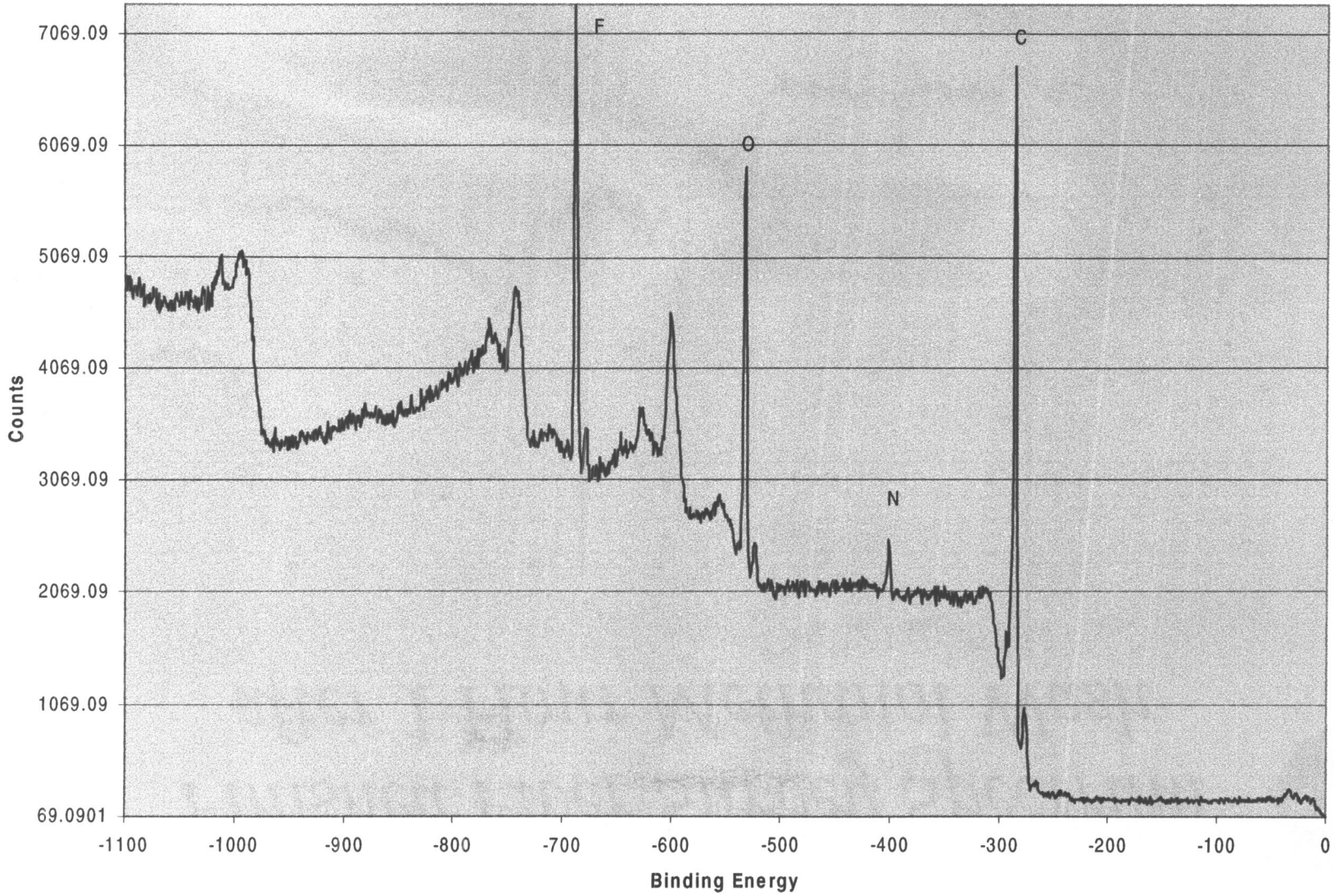
R, R' = proprietary functional groups

2. PMR-II-50 Polyimide (3.0%)
3. Acetone

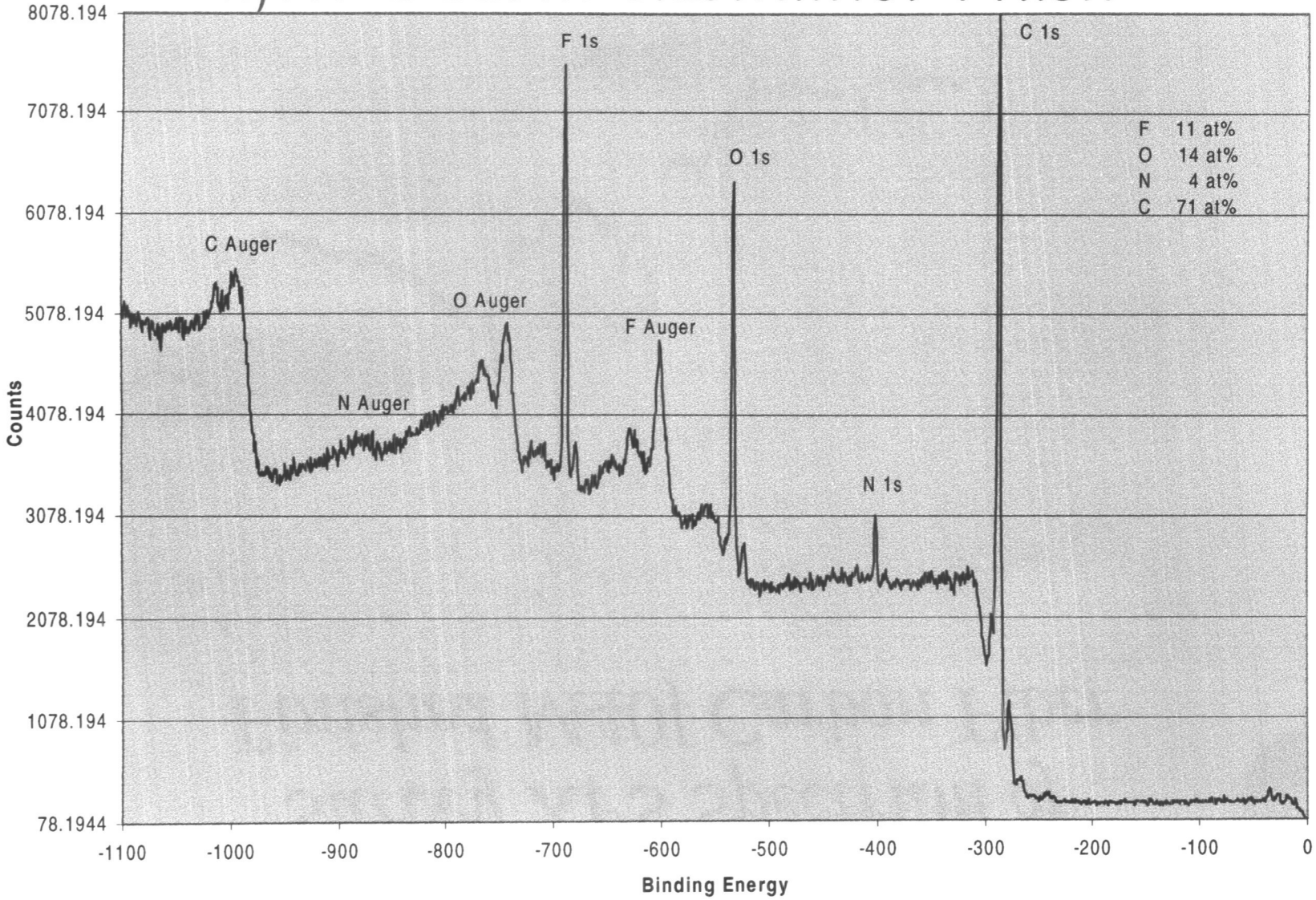
Appearance of Finished M40J Carbon Fiber



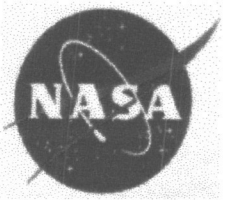
Survey XPS Spectrum of Finished M40J Carbon Fiber



Finished Fiber Survey Spectrum after 1 Hour Methanol Wash



XPS Elemental Analysis Finished M40J Carbon Fibers



Element	% As Finished	%After MeOH Wash	PMR-II-50
F	12.6	11.0	18.1
O	16.2	14.0	10.4
N	2.9	4.0	5.5
C	68.4	71.0	66.1

Mechanical Properties of M40J Tows

Test Specimen		Denier (g/9000m)		Dry Tow Tensile Testing*					Toray Data**	
Fiber Type	Conditioning	Calc. (Toray data)	Measured	Max. Load Kg	Tenacity/Tensile Strength g/d	ksi***	% drop	Strain-to Failure, %	Strength ksi	Failure Strain %
M40JB 6K	As-received/Control	**2025**	2048	57 ±6	12.8 ±1.4	290 ±32		0.7 ±0.1	650	1.20
	De-sized		**1629**	37 ±3	10.2 ±0.7	231 ±16	20%	0.6 ±0.0		
	De-sized+Re-finished		**2016**	47 ±5	10.6 ±1.1	240 ±25	17%	0.7 ±0.1		
M60JB 6K	As-received/Control	**1856**	1597	44 ±4	10.8 ±1.1	245 ±25	16%	0.6 ±0.0	590	0.70

* ASTM D885; 10 in nip-to-nip gage length; 12 in/min Cross-head Speed; Untwisted; 10 repeat tests for the mean values and standard

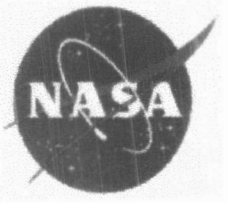
** TY-0030B-01: Properties of tow which has been resin-impregnated and then cured w/ Bakelite ERL 4221 epoxy resin fr Union Carbide.

*** Conversion formular from g/d to psi: psi = g/d x density x 12,791

FY 01 Conclusions

- Addition of ultrasound to continuous desizing line reduced residual size to a few submicron particles
- M40J fiber structure highly irregular with significant (10-15%) oxygen
- Reactive finish containing PMR-II-50 polyimide coats fibers uniformly and chemically bonds to the fiber surface

Program Status and Future Work



- Quantities of desized and desized/finished M40J fibers produced for unicomposite fabrication
- In FY 02, thermomechanical and moisture resistance testing will be conducted
- Fiber treatment to be optimized based on program results
- Fabric composites to be evaluated with similar approach

PROPERTIES OF M40J CARBON/PMR-II-50 COMPOSITES FABRICATED WITH DESIZED AND SURFACE TREATED FIBERS

**Ronald E. Allred*, Jan M. Gosau*,
E. Eugene Shin**, Linda S. McCorkle**, and James K. Sutter**,
Michelle O'Malley***Abstract**

To increase performance and durability of high temperature composites for potential rocket engine components, it is necessary to optimize wetting and interfacial bonding between high modulus carbon fibers and high temperature polyimide resins. It has been previously demonstrated that the electro-oxidative shear treatments used by fiber manufacturers are not effective on higher modulus fibers that have fewer edge and defect sites in the surface crystallites. In addition, sizings commercially supplied on most carbon fibers are not compatible with polyimides. This study was an extension of prior work characterizing the surface chemistry and energy of high modulus carbon fibers (M40J and M60J, Torray) with typical fluorinated polyimide resins, such as PMR-II-50 . . A continuous desizing system which utilizes environmentally friendly chemical-mechanical processes was developed for tow level fiber and the processes were optimized based on weight loss behavior, surface elemental composition (XPS) and morphology (FE-SEM) analyses, and residual tow strength of the fiber, and the similar approaches have been applied on carbon fabrics. Both desized and further treated with a reactive finish were investigated for the composite reinforcement. The effects of desizing and/or subsequent surface re-treatment on carbon fiber on composite properties and performance including fiber-matrix interfacial mechanical properties, thermal properties and blistering onset behavior will be discussed in this presentation.

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