ornl

Manufacturing of Carbon Fibers Using Microwave Assisted Plasma Technology

*Managed and operated by UT-Battelle, LLC for the U.S. Department of Energy under contract DE-AC05-00OR22725.

Objective

- To develop a microwave assisted technical alternative to carbonize and graphitize polyacrylonitrile (PAN) based precursor.

Introduction

- Role/purpose in the overall Partnership for a New Generation of Vehicles (PNGV) Program.
- Lightweight materials:
  - Weight reduction.
  - Carbon fiber composites.

Carbon Fibers

- High interest by automotive manufacturers in developing carbon fiber based composites for structural applications.
- Carbon fiber composites can reduce automobile weight by 30 to 50 %.
- Weight reduction: fuel consumption, better fuel economy, emission reductions.
- **High Cost!!**

Carbon Fiber Cost Reduction Options

- Scale-up of traditional production system (requires no technological breakthrough).
- Drastic price reduction in the precursor fiber for traditional production system.
- Technological breakthrough.

Critical Need

- A manufacturing breakthrough is required for carbon fiber to reach its full potential as a structural material in the automotive and commercial world.
- **Carbon fiber cost reduction is essential.**

Goals

- Economic: from actual \$8.00/lb to equal or less \$5.00/lb
- Mechanical properties:
  - Ultimate tensile strength: 300 – 400 ksi
  - Young's Modulus: 25 Msi
  - Elongation at failure: >1%
- Most recent information

Technical Feasibility

- Initial energy source: Variable Microwave Frequency Energy (VFME).
- Fiber fingerprint properties: base line data for comparison is needed.
- Development of testing/processing infrastructures.
- Batch processing.

Fiber Characterization and Fingerprint Properties

- Density: bulk & intrinsic (pycnometer) at various stages.
- Electrical resistivity.
- Fiber diameter.
- Mechanical evaluations.
- Network analysis: dielectric properties.
- X-ray diffractometry.

Electrical Resistivity

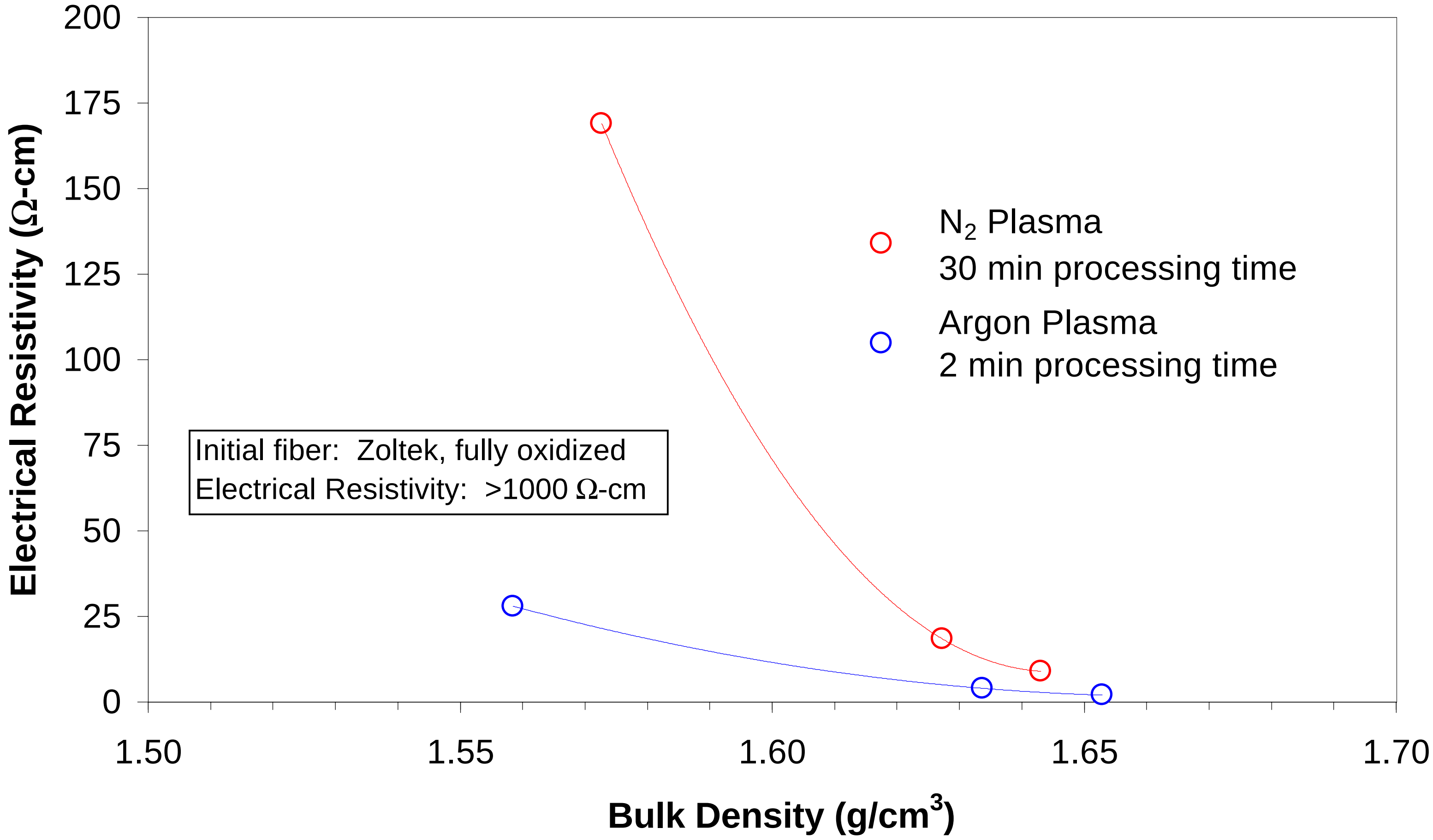
Conductors		
Silver	1.5E-6	Ω-cm
Copper	1.7E-6	Ω-cm
Aluminum	2.6E-6	Ω-cm
Iron	10.0E-6	Ω-cm
Stainless Steel	50.0E-6	Ω-cm
Insulators	>1E+10	Ω-cm
Semi-conductors	1E-2 to 1E+7	Ω-cm
Graphitized Carbon Fibers	2E-4 to 2E-3	Ω-cm
Oxidized PAN Precursor	>1000	Ω-cm

Technical Feasibility, Fiber Processing and Their Results

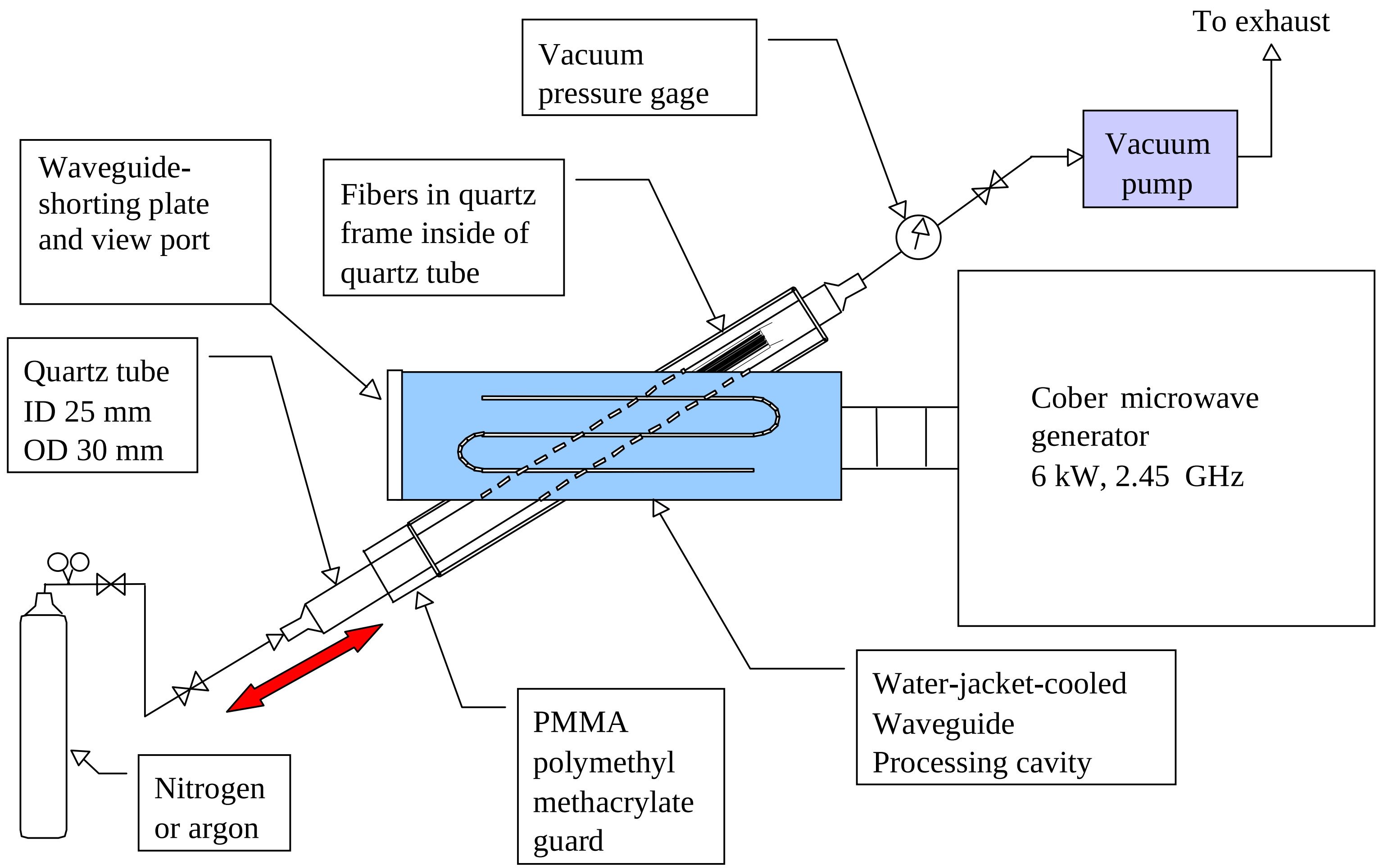
- VFME: did not produce positive results, and was discarded as a primary energy source for this process.
- Single frequency microwave energy became a major player.
- Fiber fingerprint properties were obtained.
- Fiber testing infrastructures were developed.
- Microwave - assisted plasma technology.

Results

- Feasibility demonstrated.
- Quasi-batch process.
- Start material/precursor: stabilized or partially stabilized PAN-fiber.
- Testing of produced samples.
- Parametric studies. (Obtainment of processing data.)

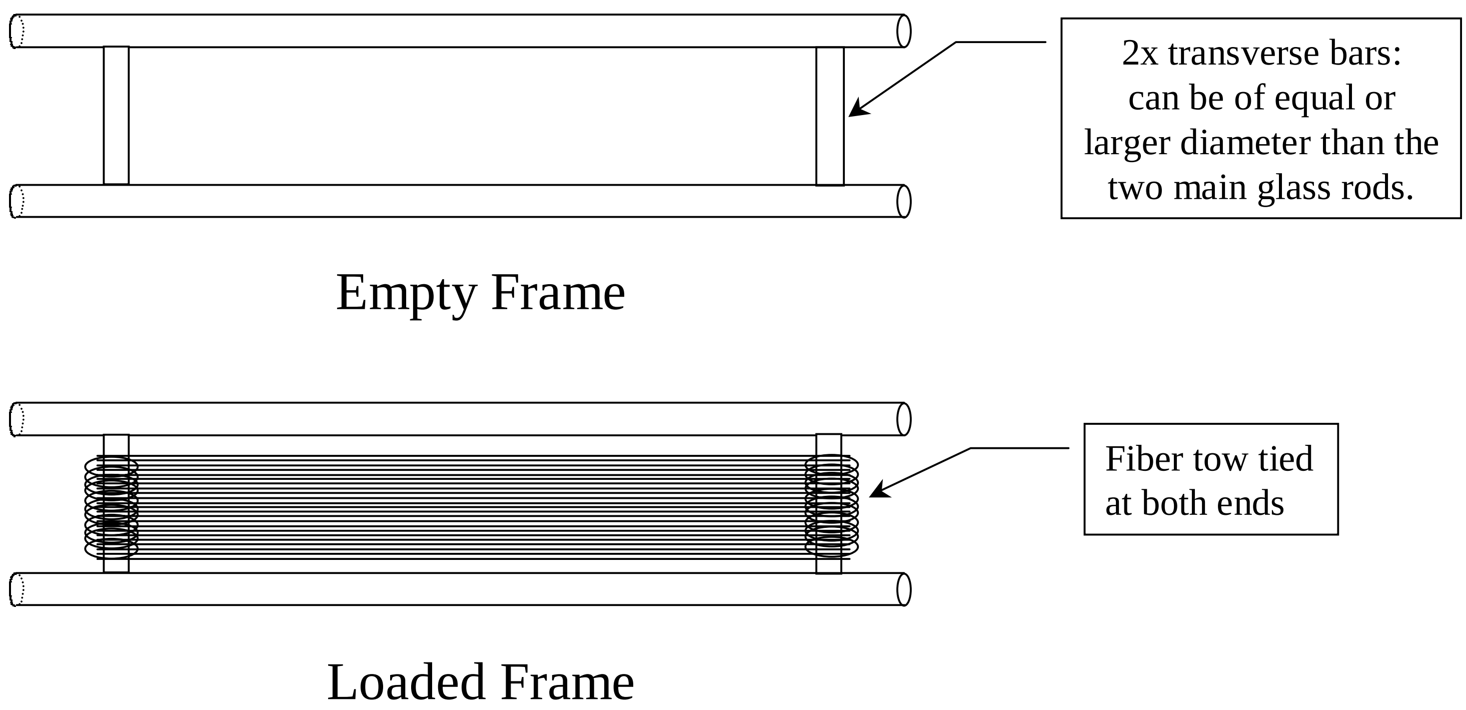


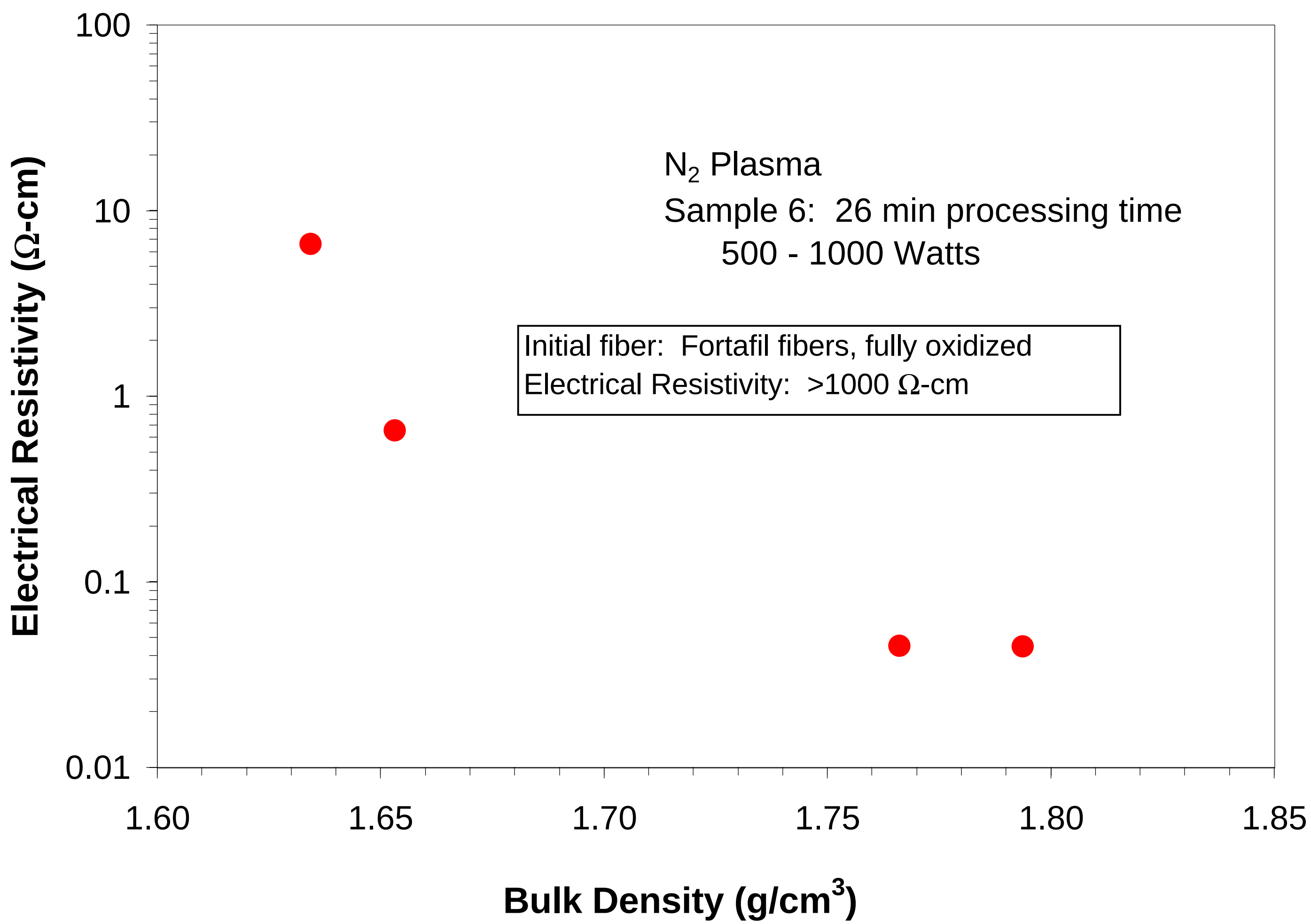
Electrical resistivity as a function of bulk density for plasma processing of several short samples



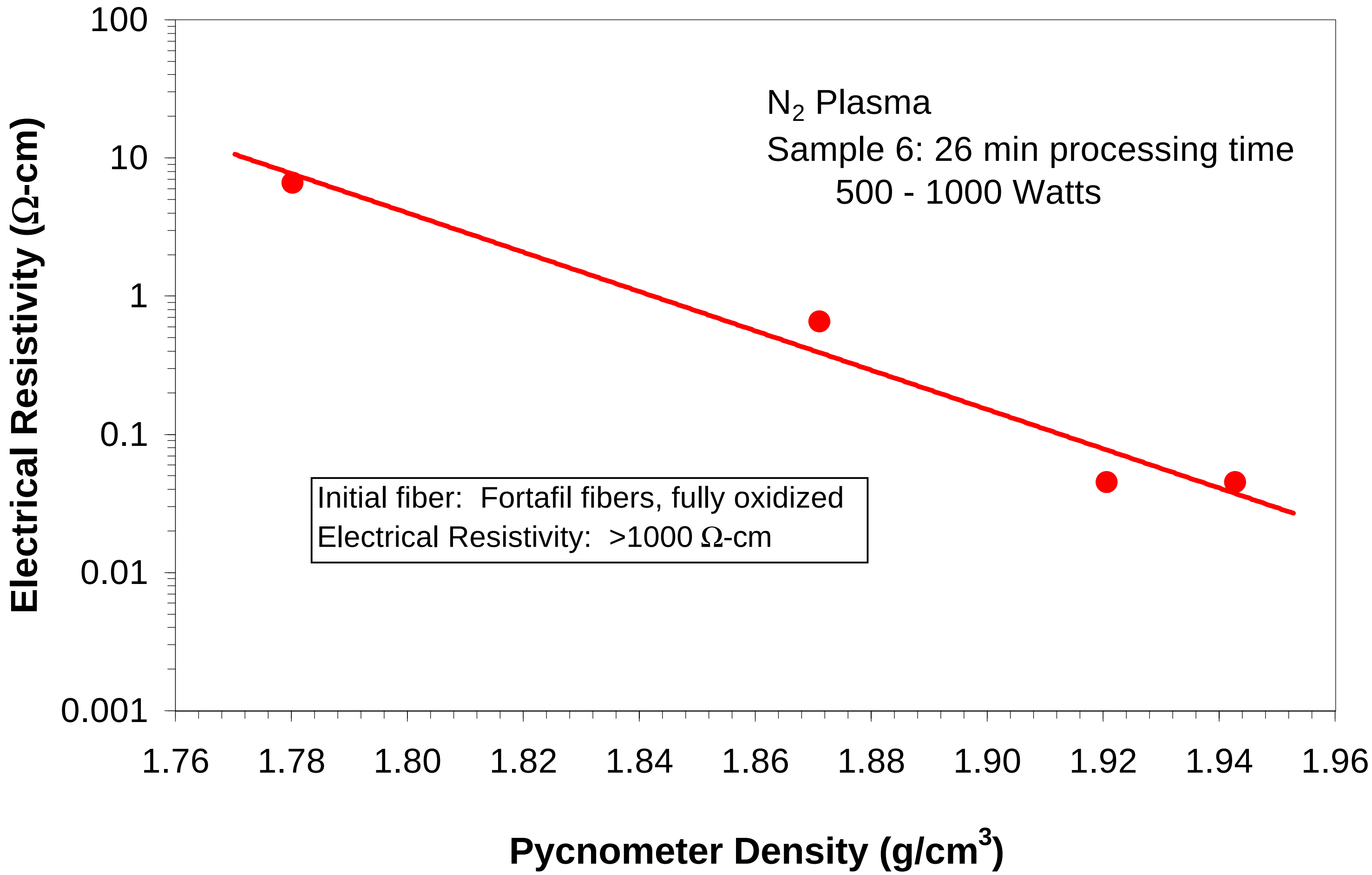
Demonstration of fiber processing via microwave plasma

Fiber Sample Holder - Double H Frame

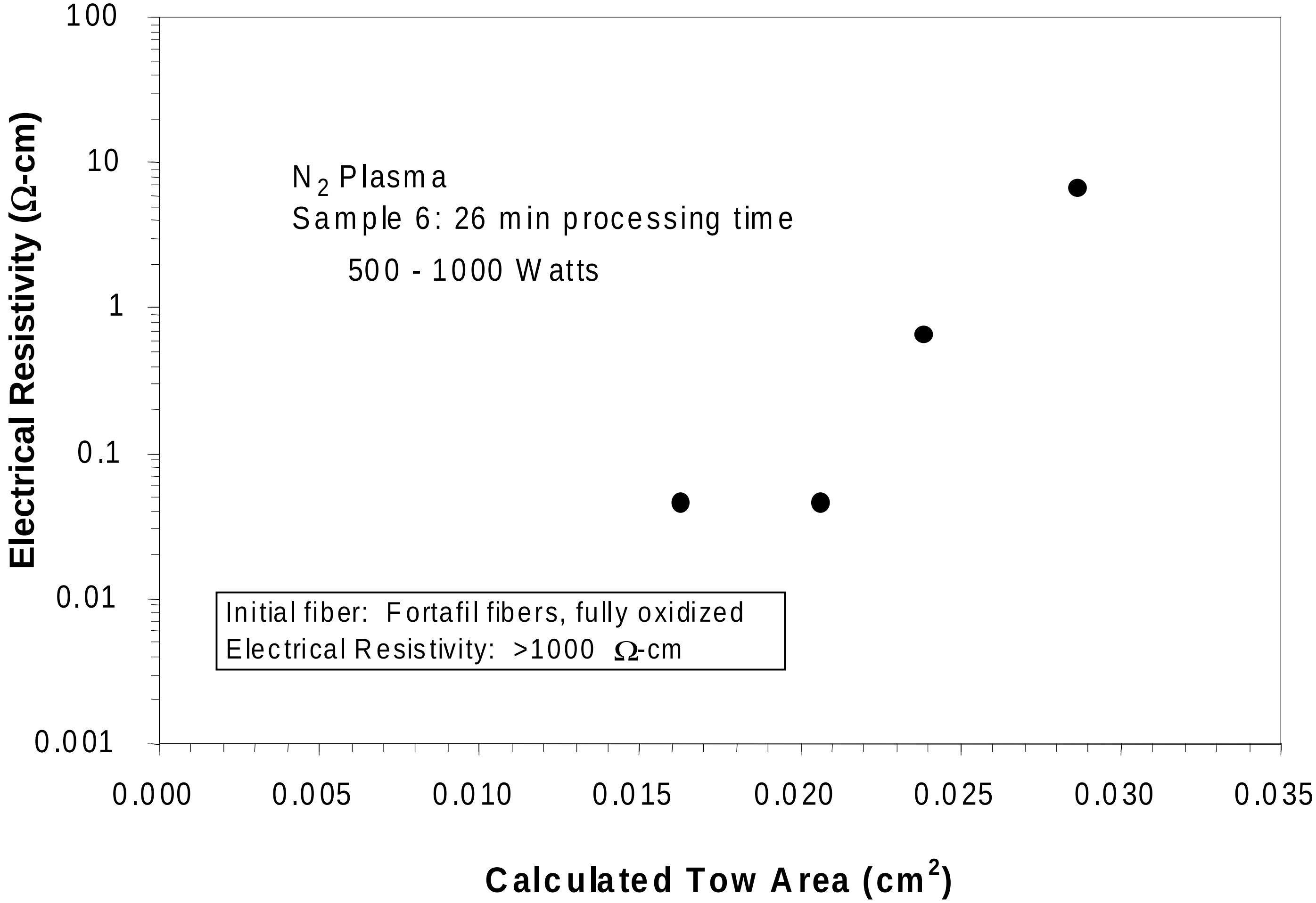




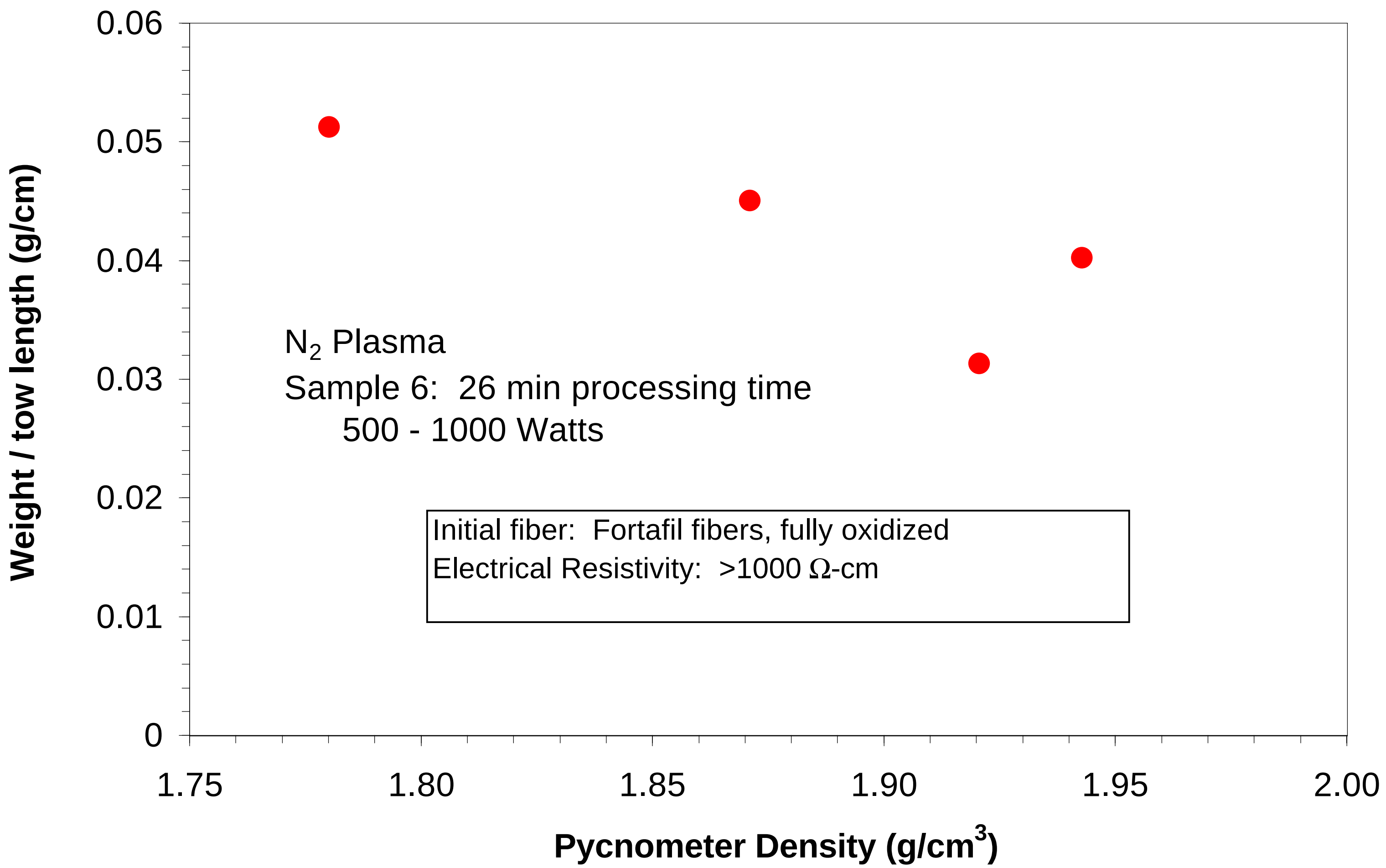
Electrical resistivity as a function of bulk density for plasma processing of sample 6



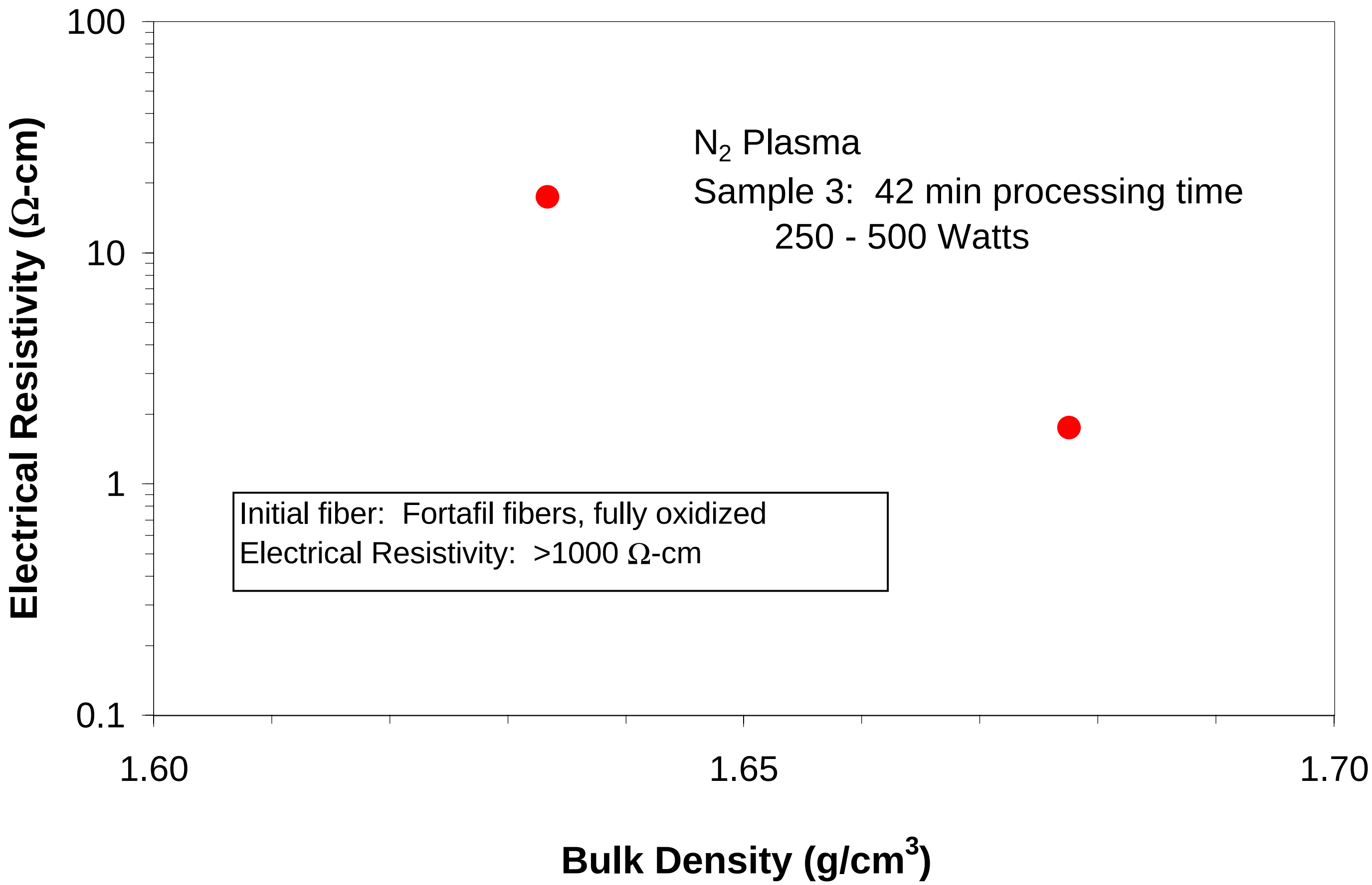
Electrical resistivity as a function of pycnometer density for plasma processing of sample 6



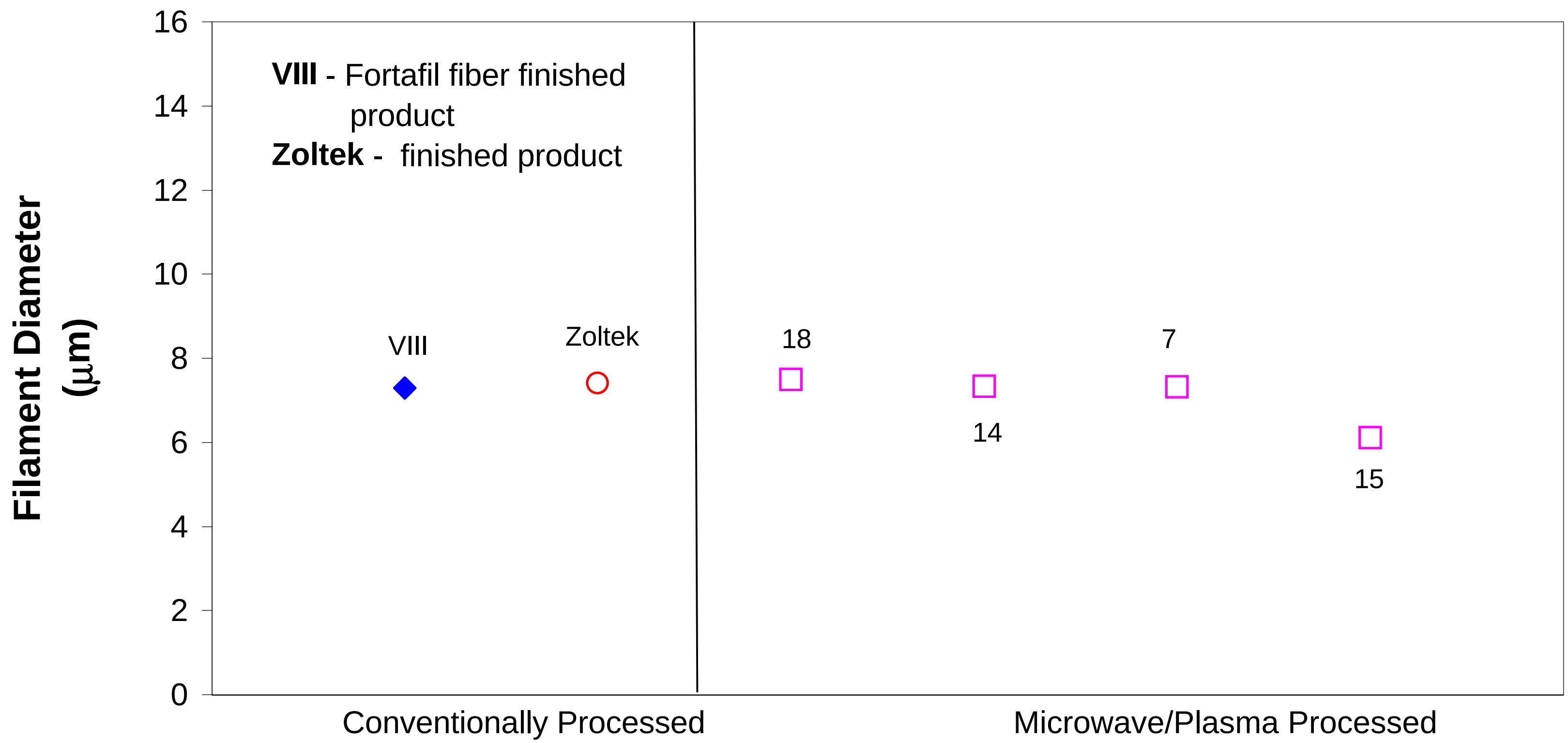
Electrical resistivity as a function of calculated tow area for plasma processing of sample 6



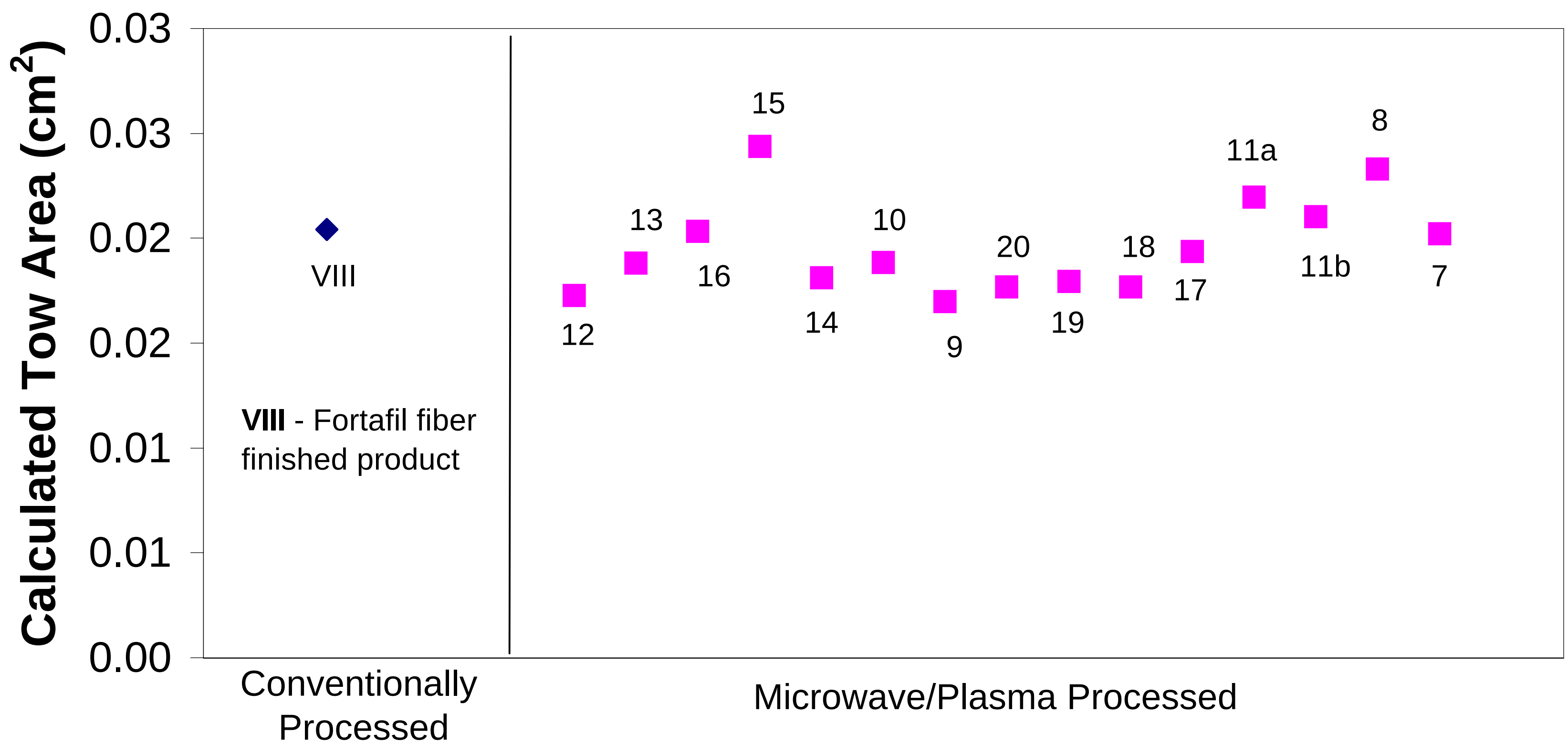
Weight/tow length as a function of pyconometer density for plasma processing of sample 6



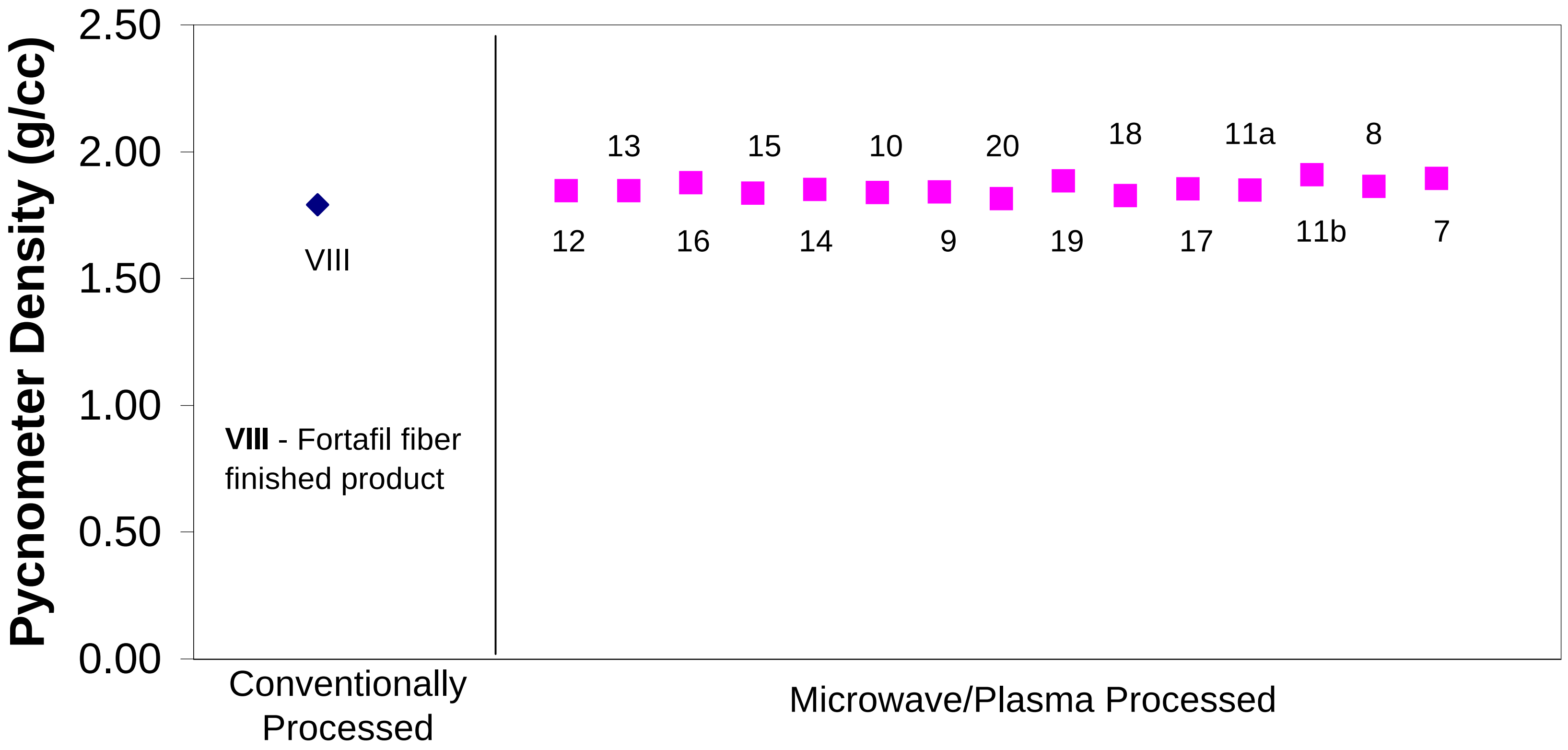
Electrical resistivity as a function of bulk density for plasma processing of sample 3



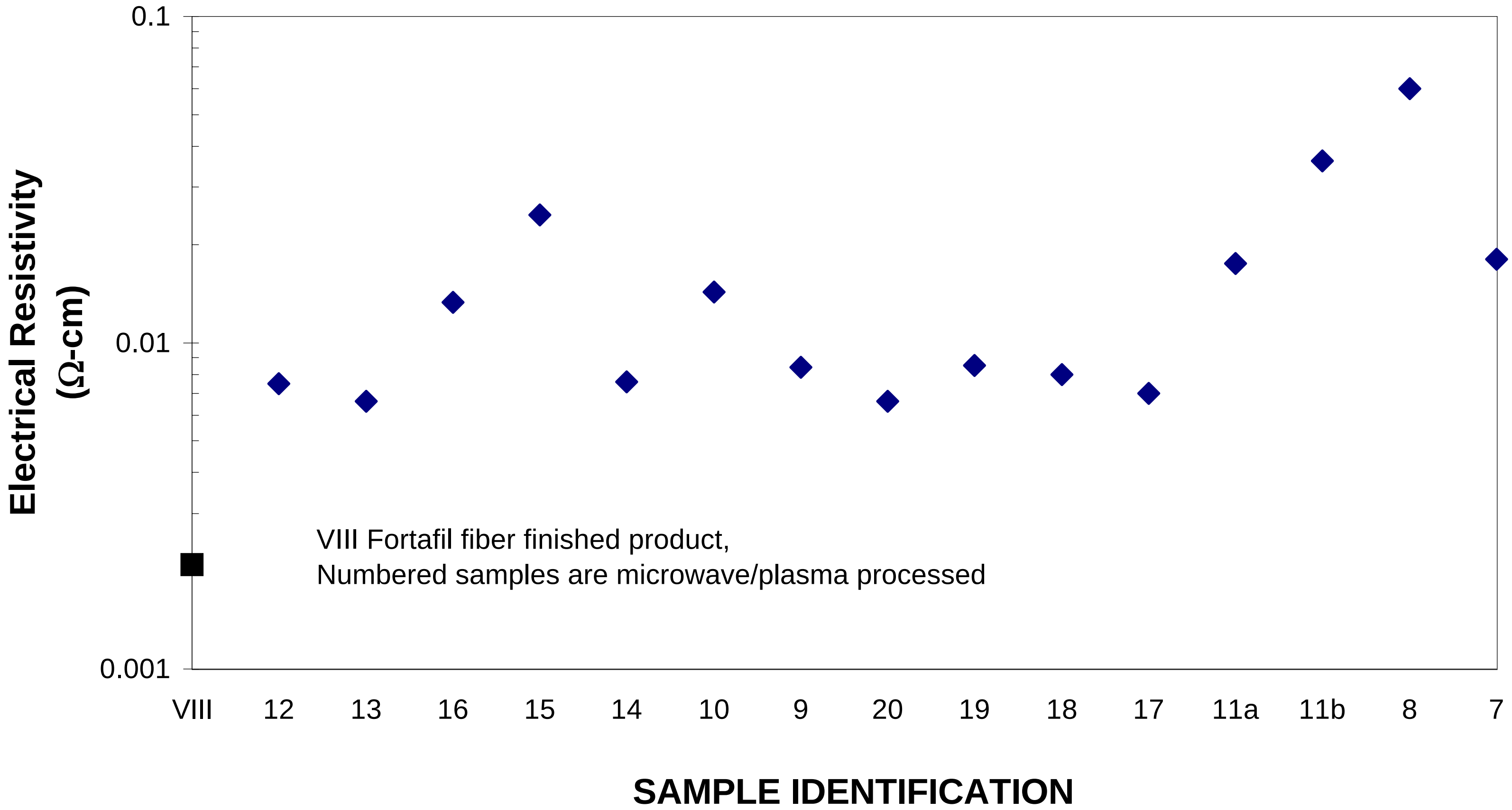
Comparison of fiber diameters of conventionally processed and plasma processed fibers



Comparison of the calculated fiber tow areas of conventionally processed and plasma processed fibers



Comparison of pycnometer density of conventionally processed and plasma processed fibers



Comparison of electrical resistivity of conventionally processed and plasma processed fibers

Mechanical Testing Data

Fibers tested after graphitization without surface treatments or sizing.

PAN - precursor based fiber, 50K tows

	Conventionally graphitized	**Microwaved/ plasma processed**
Ultimate Tensile Strength, σ_B (ksi)	443.1	203 - 344
Young's Modulus, E (Msi)	29.16 - 30.0	16.3 - 27.91
Elongation at Failure, ε (%)	1.27	0.73 - 0.95

Economic Evaluation

- A cost assessment comparison between conventional & microwave/plasma carbon fiber manufacturing technologies was performed.
- Assessment base on a MIT model, correspondently modified for this task.
- This assessment was sent to different carbon fiber manufacturers for their evaluation and comments.
- Carbon fiber manufacturers validated the model accuracy for conventional manufacturing.
- Model indicates that the overall cost reduction via microwave/plasma technology will be around 19%.

Beneficial Effects

- Effective processing time (from oxidized or partially oxidized to graphitized and plasma treatment) has been reduced substantially. Effective total processing time: 5 - 8 min.
- Replace part of the oxidization/stabilization, carbonization, and graphitization stages.
- Plasma fiber surface treatment can be performed as part of processing.
- Lowers capital investment.

Beneficial Effects Continued

- Unique tailorable properties (important issue).
- Fast start-up/shut-down times.
- Quicker response control.

Future Actions

- Multiple patents have been filed or disclosed, more are expected.
- From batch (Quasi-Batch) processing to continuous processing (in progress).
- Further evaluation in plasma based fiber surface treatments.

Potential Spin-offs

- Further advancements in plasma based fiber surface treatments. Tailor surface energy of carbon fiber.
- Carbon fiber reactivation:
  - Recycling.
  - Fiber surface customization.
- Mechanical interlocking functionability.
- Real time, diagnostic monitoring of carbon fiber during the manufacturing process.