NASA/TM—2014-218349

Status and Evaluation of Microwave Furnace Capabilities at NASA Glenn Research Center

Maricela Lizcano
Glenn Research Center, Cleveland, Ohio

Jonathan A. Mackey
University of Akron, Akron, Ohio

November 2014

NASA STI Program . . . in Profile

Since its founding, NASA has been dedicated to the advancement of aeronautics and space science. The NASA Scientific and Technical Information (STI) program plays a key part in helping NASA maintain this important role.

The NASA STI Program operates under the auspices of the Agency Chief Information Officer. It collects, organizes, provides for archiving, and disseminates NASA's STI. The NASA STI program provides access to the NASA Aeronautics and Space Database and its public interface, the NASA Technical Reports Server, thus providing one of the largest collections of aeronautical and space science STI in the world. Results are published in both non-NASA channels and by NASA in the NASA STI Report Series, which includes the following report types:

- TECHNICAL PUBLICATION. Reports of completed research or a major significant phase of research that present the results of NASA programs and include extensive data or theoretical analysis. Includes compilations of significant scientific and technical data and information deemed to be of continuing reference value. NASA counterpart of peer-reviewed formal professional papers but has less stringent limitations on manuscript length and extent of graphic presentations.

- TECHNICAL MEMORANDUM. Scientific and technical findings that are preliminary or of specialized interest, e.g., quick release reports, working papers, and bibliographies that contain minimal annotation. Does not contain extensive analysis.

- CONTRACTOR REPORT. Scientific and technical findings by NASA-sponsored contractors and grantees.

- CONFERENCE PUBLICATION. Collected papers from scientific and technical conferences, symposia, seminars, or other meetings sponsored or cosponsored by NASA.

- SPECIAL PUBLICATION. Scientific, technical, or historical information from NASA programs, projects, and missions, often concerned with subjects having substantial public interest.

- TECHNICAL TRANSLATION. English-language translations of foreign scientific and technical material pertinent to NASA's mission.

Specialized services also include creating custom thesauri, building customized databases, organizing and publishing research results.

For more information about the NASA STI program, see the following:

- Access the NASA STI program home page at *http://www.sti.nasa.gov*

- E-mail your question to *help@sti.nasa.gov*

- Fax your question to the NASA STI Information Desk at 443–757–5803

- Phone the NASA STI Information Desk at 443–757–5802

- Write to:
  STI Information Desk
  NASA Center for AeroSpace Information
  7115 Standard Drive
  Hanover, MD 21076–1320

NASA/TM—2014-218349

Status and Evaluation of Microwave Furnace Capabilities at NASA Glenn Research Center

Maricela Lizcano
Glenn Research Center, Cleveland, Ohio

Jonathan A. Mackey
University of Akron, Akron, Ohio

National Aeronautics and
Space Administration

Glenn Research Center
Cleveland, Ohio 44135

November 2014

This report contains preliminary findings, subject to revision as analysis proceeds.

Level of Review: This material has been technically reviewed by technical management.

Available from

NASA Center for Aerospace Information
7115 Standard Drive
Hanover, MD 21076–1320

National Technical Information Service
5301 Shawnee Road
Alexandria, VA 22312

Available electronically at http://www.sti.nasa.gov

Status and Evaluation of Microwave Furnace Capabilities at NASA Glenn Research Center

Maricela Lizcano
National Aeronautics and Space Administration
Glenn Research Center
Cleveland, Ohio 44135

Jonathan A. Mackey
University of Akron
Akron, Ohio 44325

Abstract

The microwave (MW) furnace is a HY-Tech Microwave Systems, 2 kW 2.45 GHz Single Mode Microwave Applicator operating in continuous wave (CW) with variable power. It is located in Cleveland, Ohio at NASA Glenn Research Center. Until recently, the furnace capabilities had not been fully realized due to unknown failure that subsequently damaged critical furnace components. Although the causes of the problems were unknown, an assessment of the furnace itself indicated operational failure may have been partially caused by power quality. This report summarizes the status of the MW furnace and evaluates its capabilities in materials processing.

Status

The MW furnace and the lab in which it is housed have been fully upgraded. The MW furnace was upgraded with the installation of a new isolator and circulator (Gerling Applied Engineering Inc., GAE, Modesto, CA). The power supply and the magnetron were sent to GAE for repair and returned. Additionally, in the event that an arc is discharged within the length of the waveguide during operation, GAE installed an arc detector in the magnetron that shuts power off and prevents damage to the components such as the isolator, circulator, magnetron or power supply. To address potential problems with power quality, a new power line and electrical receptacle were installed in the lab. A double-conversion Universal Power Supply (UPS) and power conditioner were also implemented. The MW power supply, the water cooler and computer are directly plugged into the conditioner and UPS. The MW furnace is configured to operate under a controlled atmosphere and vacuum. Since these upgrades and modifications have been implemented, the status of the MW furnace is operational as of May 2013 with no other failures noted to date.

One additional upgrade/repair needs to be completed. The furnace pyrometer seems to be working but data acquisition is not possible. It is unclear whether the problem is related to software, cabling or the pyrometer itself. Several attempts to repair this problem have been unsuccessful. Precise monitoring of temperature is an important part of material processing. It is recommended that a new temperature monitoring system be purchased and implemented for controlled material processing experiments. Two systems are currently being evaluated: (1) IR pyrometer or (2) IR Camera. A supplementary report with a recommendation for a temperature acquisition system will be submitted at a later date.

Evaluation of Capabilities

The MW furnace was utilized in various experiments. The in-house MW furnace has demonstrated capabilities in materials alloying, melting, sintering, nanofiber growth, and conversion of organic-inorganic polymer to ceramic material. The following paragraphs describe these experiments.

Alloying

The MW furnace has been demonstrated to be an effective processing tool for the alloying of thermoelectric powders. Thermoelectric composites consisting of a Si/Ge matrix with WSi_2 nano-inclusions is desired. Direct mechanical alloying of the powders, in a planetary mill, was found to suitably alloy the Si/Ge matrix phase but could not react the W. A short 15 min exposure to microwave energy has been found to react the elemental W into the desired WSi_2 phase. Figure 1 shows the XRD of the starting powder as obtained from mechanical alloying (black curve). The starting powder consists of an alloyed Si/Ge phase with an unreacted W phase. After 5 min at 500 W (orange curve) some of the W is reacted into a WSi_2 phase. Exposure to 700 W for 15 min (green curve) consumes the entire W content to form the desired WSi_2 phase. The final product of the experiment was a powder suitable for hot pressing or sintering by other means. The broad XRD reflections in the starting powder indicate nano-sized crystallites, while the sharp peaks after the MW heat treatment indicate crystallite growth.

Melting and Sintering

Systems with low processing temperatures such as Bi or Sb based skutterudite thermoelectrics can be easily sintered or melted with the MW system. Samples composed predominantly of Bi, melting point 270 °C, were melted, and solidified in quartz (SiO_2) crucibles within the MW cavity. The Bi based samples are found to directly couple with the microwave energy, these samples do not require a susceptor. The ingots release from the SiO_2 crucibles and were easily machined into a number of samples required for thermoelectric characterization, Figure 2.

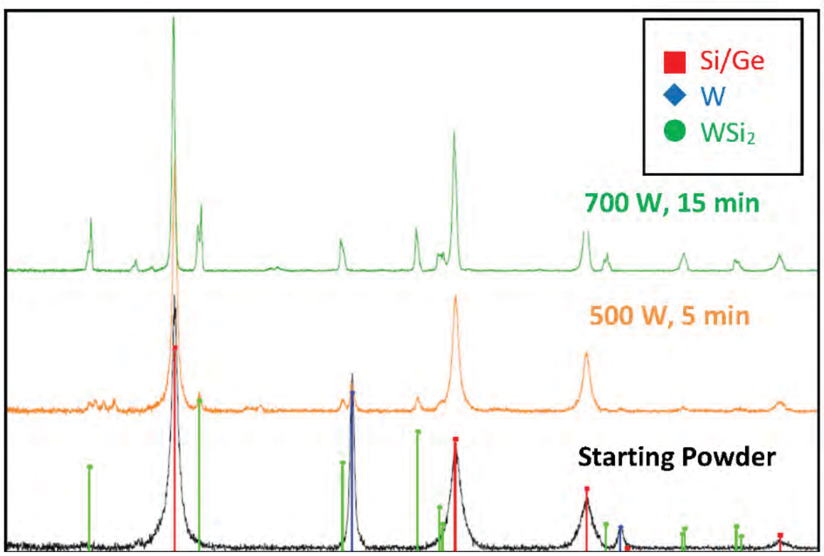


Figure 1.—Demonstration of microwave alloying of powders.

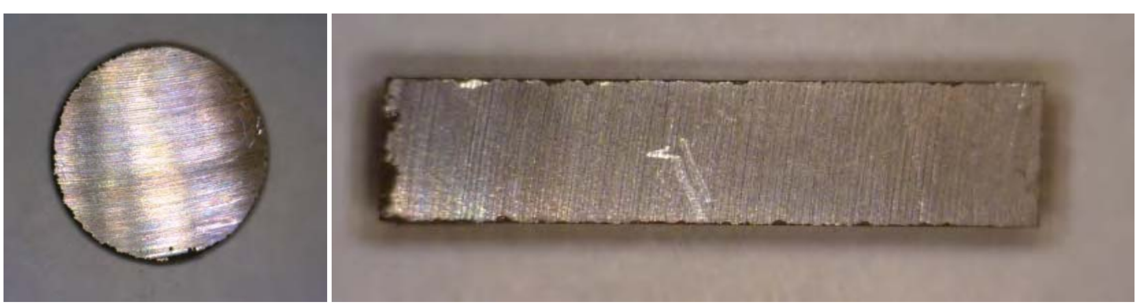


Figure 2.—Ni-Bi-Ge thermoelectrics prepared with microwave sintering.

Sintering of higher temperature materials like Si/Ge (melting range 937 to 1413 °C) has been attempted. The samples are found to directly couple with the MW radiation, and can reach stable temperatures in excess of 900 °C. At this time, the samples have not been successfully sintered to a suitable density. Instead local heating leads to melting identified as the spherical solidified phase in Figure 3. The local melting of the samples occurs before the bulk has the opportunity to sinter. Further experimental work needs to be conducted to prevent local heating and promote homogenous heating.

A preliminary experiment was conducted to determine feasibility of an alternate sintering route for Aeronautical Sciences (AS) advanced SiC fiber development. A 1 1/2 in. tow was cut from a Sylramic spool and placed inside a small graphite tube. The sample was then placed vertically inside the furnace. A quartz tube was placed over the entire sample to control processing atmosphere. The Sylramic tow was irradiated for 20 min. Figure 4 shows an SEM image of (a) and (b) Sylramic (as received) fiber (c) and (d) Sylramic fiber that has been MW irradiated. Reduced porosity in the center of the fiber is noted for the fibers exposed to MW irradiation. In particularly, a reduction in pores ≤ 50 nm can be observed (when comparing image (b) and (d). Additionally, a more uniform microstructure is observed which may provide an avenue to reduce core/shell effect often seen in in-house sintered SiC fibers.

Figure 3.—Left: view of Si/Ge sample sintering in microwave. Right: final Si/Ge sintered sample, local heating leads to non-uniform melting of the sample.

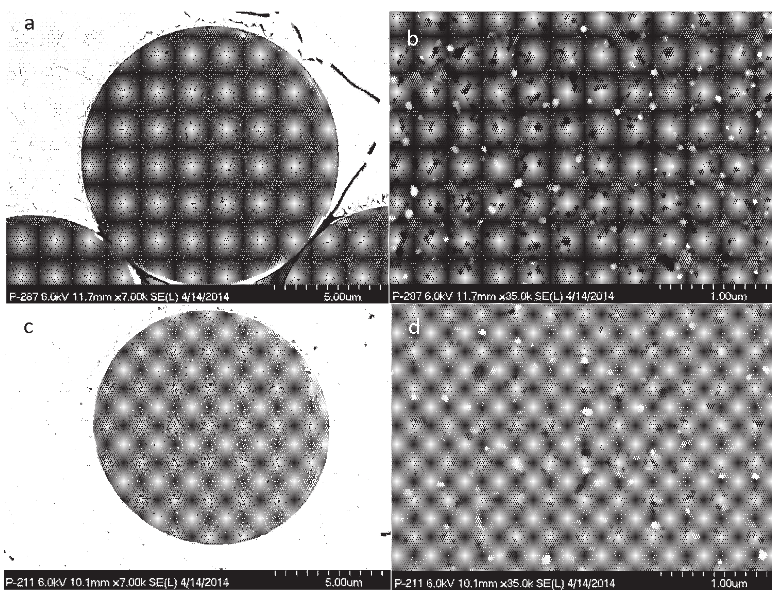


Figure 4.—SEM images of Sylramic fiber (a) and (b) as received and MW treated Sylramic fiber image (c) and (d).

Nanofiber Fabrication

Rapid growth of boron nitride (BN) nanofibers has been demonstrated with the MW furnace (Fig. 5). The BN nanofibers were grown from a BN stage in less than 1/2 hr. The morphology of the final BN consists of 1 μm diameter bundles of 100 nm fibers. Figure 4 shows the BN nanofibers at three magnification levels.

Polymer Derived Ceramics (PDC)

The MW furnace was used to convert an organic-inorganic polymer material into ceramic SiC nanofibers demonstrating this capability. The spun pre-ceramic nanofibers were made from a polymer blend. The fibers were placed between two susceptor plates inside the MW chamber. The power was ramped up at 100 W intervals every 3 min, with an additional 3 min dwell time at 600 W. Temperatures of ~1140 °C were observed during the dwell time. The XRD result is shown in Figure 6. The peaks indicate conversion of precursor nanofibers to β-SiC nanofibers with 2θ = 35.62°, 41.42°, 59.95° and 71.74° ascribed to lattice planes (111), (200), (220), and (311), respectively. Figure 7 shows SEM images of (a) preceramic nanofibers, and (b) and (c) the MW converted ceramic SiC fibers. The uniform microstructure in the images demonstrates the advantage of MW irradiation as a rapid processing avenue for PDCs.

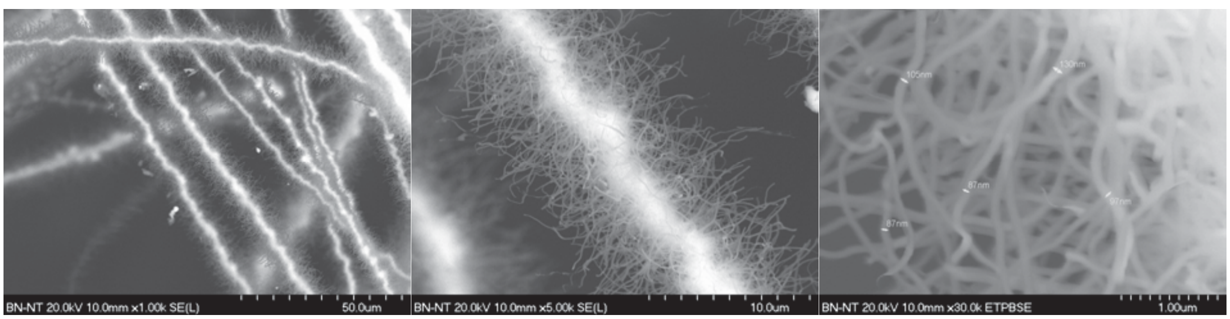


Figure 5.—Demonstration of BN nanofibers rapidly grown in a microwave cavity. Final BN product consists of 1 μm diameter bundles of 100 nm fibers.

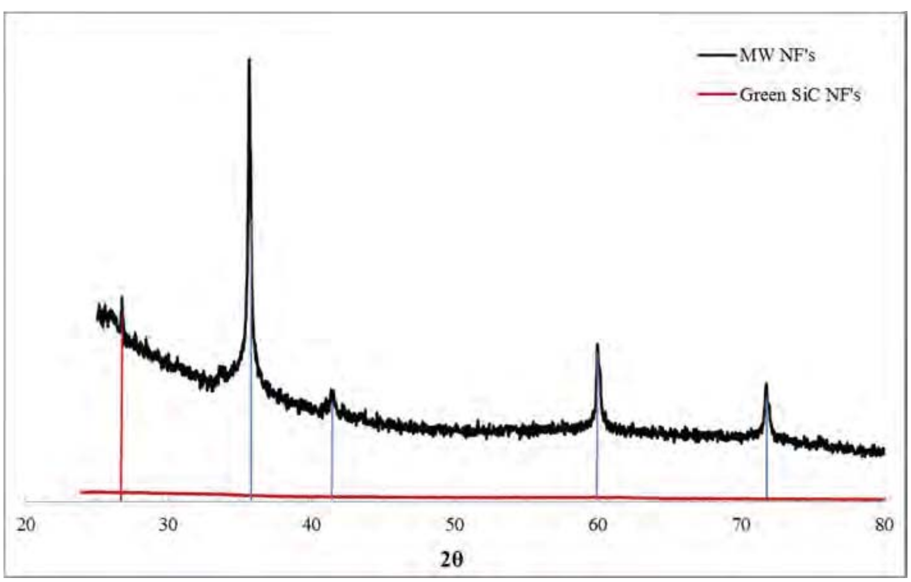


Figure 6.—XRD of precursor nanofibers and MW energy converted nanofibers.

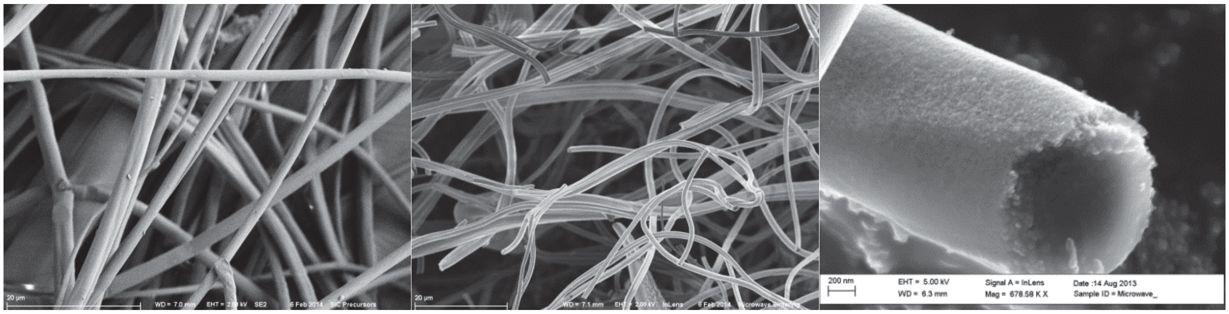


Figure 7.—SEM images of (a) preceramic nanofibers, (b) and (c) MW ceramic converted SiC nanofibers.

Summary

The 2.45 GHz single-mode microwave furnace located at NASA Glenn Research Center has been upgraded and is operational. The furnace is fitted with a quartz tube and vacuum for environmental control during operation. Variable power output is in the range of 100 W to 2 kW. The unit has demonstrated capabilities in materials processing such as alloying, melting, sintering, nanofiber fabrication and ceramic conversion of polymeric materials.

National Aeronautics and Space Administration

APPENDIX 1

www.nasa.gov 1

National Aeronautics and Space Administration

A Preliminary Study in The Use of Microwave Energy To Produce Microstructure Uniformity in SiC Tows,

Maricela Lizcano, PhD

NASA Glenn Research Center

04/24/2014

www.nasa.gov

National Aeronautics and Space Administration

Objective and Task

Introduction: Microwave sintering offers an advantage over conventional heating due to rapid molecular heating as opposed to convection and conduction heating using an electric furnace. This translates to reduced heating temperatures as well as reduced heating times and a cost saving route for SiC tow processing. It is expected that uniform heating will produce uniform microstructures.

Objective: Determine feasibility of alternate sintering route for UHT SiC fiber development.

Task: Use microwave energy to irradiate SiC (sintered) tow and determine effects on microstructure using SEM.

www.nasa.gov

National Aeronautics and Space Administration

Experimental

A 2.45 GHz microwave furnace was used during this experiment to determine the feasibility of using microwave energy for rapid sintering of SiC fiber.

A 1 1/2 in. tow was cut from a Sylramic spool and placed inside a small graphite tube. The sample was then placed vertically inside the furnace. A quartz tube was placed over the entire sample to control processing atmosphere. The sample was irradiated for 20 min.

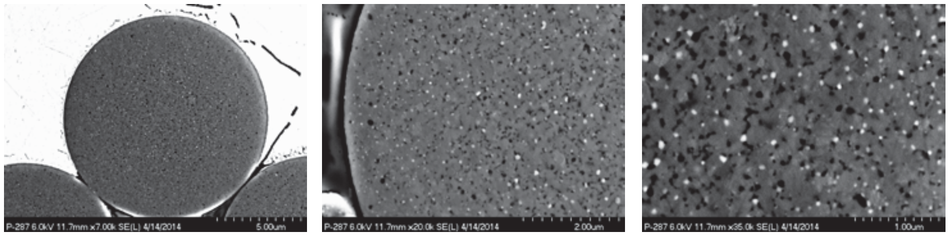
Processing Time: Start to finish, < 1 hr.

www.nasa.gov

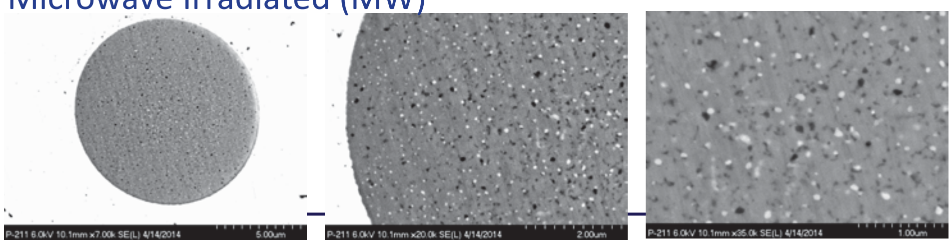
National Aeronautics and Space Administration

Preliminary Results

Sylramic (Off Spool)



Microwave Irradiated (MW)



National Aeronautics and Space Administration

A Closer Look

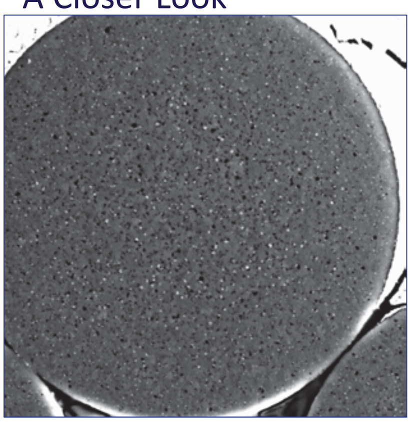


Image 1 Sylramic off Spool

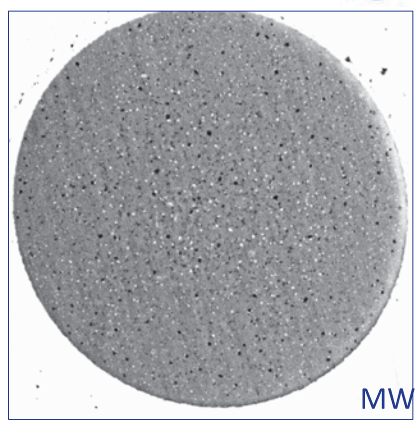


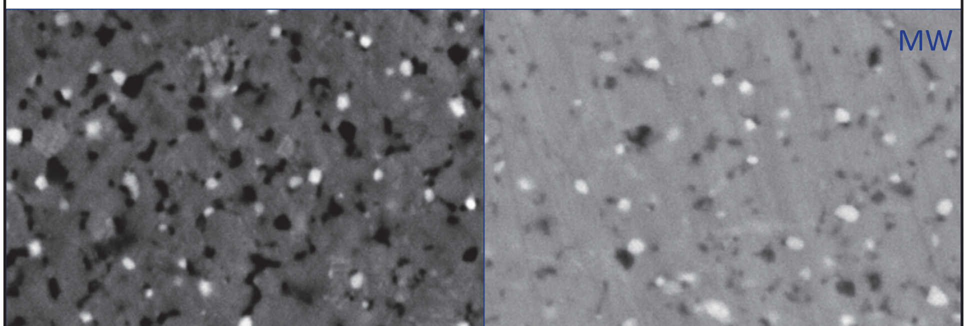
Image 2 MW treated fiber

Exposure to MW energy resulted in a more uniform microstructure with reduced porosity.

www.nasa.gov

A Closer Look

Two pore shapes and sizes are noted. Rounded pores (~100 nm) and much smaller irregularly shaped pores (≤ 50 nm). Both pore types seem to diminish throughout the cross section of the sample under MW irradiation.



Conclusions

- Microwave irradiation of a Sylramic tow shows promising results as a heat treatment method to reduce porosity and produce uniform microstructure in SiC tows.
- Further analysis of pore size and pore size distribution is necessary to confirm observations. New porometer is available on-site, however, a location for the instrument has not been determined.

National Aeronautics and Space Administration

Future Work

- Grain size evaluation

- Processing temperature data acquisition upgrade

- Strength Testing
    - Problem: current MW furnace restricts tow length that can be irradiated and then tested.
    - Solution: design a tow feed-through with step motor.

- Lox M sintering experiments.

www.nasa.gov 9