IACMI/R008-2020/6.28

Lower Cost 3D Composite Additive Manufacturing

Author: Jeff DeGrange
Date: 5 October 2020

Final Technical Report
PA16-0349-6.28.01

Approved for Public Release.
Distribution is Unlimited.

The Institute for iacmi Advanced Composites Manufacturing Innovation
THE COMPOSITES INSTITUTE

U.S. DEPARTMENT OF ENERGY

DOCUMENT AVAILABILITY

Reports produced after January 1, 1996, are generally available free via US Department of Energy (DOE) SciTech Connect.

Website http://www.osti.gov/scitech/

Reports produced before January 1, 1996, may be purchased by members of the public from the following source:

National Technical Information Service 5285 Port Royal Road
Springfield, VA 22161
Telephone 703-605-6000 (1-800-553-6847)
TDD 703-487-4639
Fax 703-605-6900
E-mail info@ntis.gov
Website http://www.ntis.gov/help/ordermethods.aspx

Reports are available to DOE employees, DOE contractors, Energy Technology Data Exchange representatives, and International Nuclear Information System representatives from the following source:

Office of Scientific and Technical Information PO Box 62
Oak Ridge, TN 37831
Telephone 865-576-8401
Fax 865-576-5728
E-mail reports@osti.gov
Website http://www.osti.gov/contact.html

Disclaimer: "The information, data, or work presented herein was funded in part by an agency of the United States Government. Neither the United States Government nor any agency thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof."

The information, data, or work presented herein was funded in part by the Office of Energy Efficiency and Renewable Energy (EERE), U.S. Department of Energy, under Award DE-EE0006926

Lower Cost 3D Composite Additive Manufacturing

Principal Investigator: Jeff DeGrange

Organization: Impossible Objects Inc

Address: 3455 Commercial Avenue, Northbrook, IL 60062

Phone: 847-400-9582 ext. 4

Email: jdegrange@impossible-objects.com

Co-authors: John Baydon PhD, John Haaland, Lisa Mueller, Brent Strong PhD

Date Published: (December 2021)

Prepared by:
Institute for Advanced Composites Manufacturing Innovation
Knoxville, TN 37932
Managed by Collaborative Composite Solutions, Inc.
For the
U.S. DEPARTMENT OF ENERGY
Under contract DE- EE0006926

Project Period:
(09/2019 – 08/2020)

Approved for Public Release

TABLE OF CONTENTS

1. LISTS

1.1 List of Acronyms

CAD:	Computer Aided Design
CBAM:	Composite Based Additive Manufacturing
CMC:	Carboxymethyl Cellulose
Flash LOI:	Flash Limiting Oxygen Index
IACMI:	Institute Advanced Composite Manufacturing Innovation
IO:	Impossible Objects
PVoH:	Poly Vinyl Alcohol
TFP:	Technical Fibre Products
Tensile CD:	Tensile Cross Direction
Tensile MD :	Tensile Machine Direction
UAMMI:	Utah Advanced Material Manufacturing Initiative
UAV:	Unmanned Air Vehicle

1.2 List of Figures

1.3 List of Tables

1.4 List of Appendices

2. EXECUTIVE SUMMARY

The purpose of the project was to find a lower cost carbon fiber material solution that would lower end use part cost produced with the Impossible Objects' 3D Composite Based Additive Manufacturing (CBAM) technology. The two strands of the research were to consider lower cost carbon tow material provided by Oak Ridge National Lab (ORNL) and carbon fiber nonwoven veils containing a soluble binder.

The CBAM additive process (Figure 1) starts with slicing a CAD file into layers and uses this digital CAD layer data to inkjet print the CAD image onto the fiber sheet, next process step is to deposit thermoplastic powder followed by stacking the sheet, printed sheets are placed into heated compression press to form the composite parts and the final step is to place the build block of consolidated carbon fiber sheets into a media blast cabinet to remove the formed composite parts from the build block. The project goal is to place the build block of compressed fiber sheets into a hot water bath while stirring to remove the formed parts from the build block and recycle the remaining unfused carbon fibers.

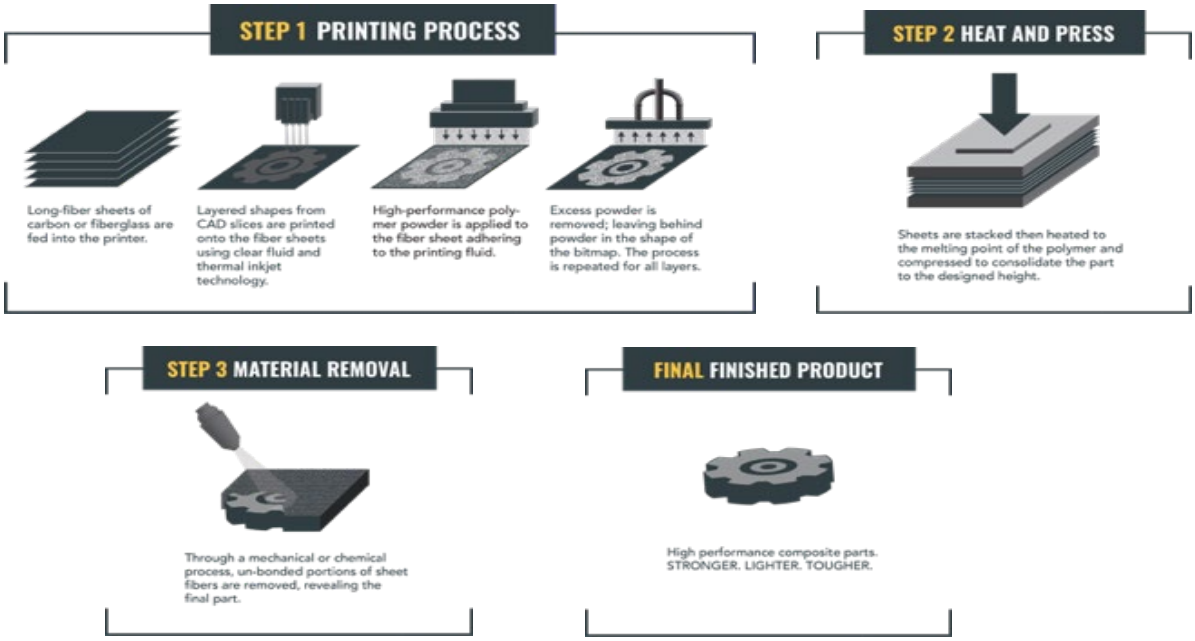


Figure 1: CBAM additive lamination process steps

The project evaluated different lower cost carbon fiber substrate options with different soluble binder types, evaluated different types of nylon 12 powder in an effort to reduce the final part cost of producing a CBAM thermoplastic parts and increase the level of recycling of the removed carbon fiber.

TFP was the project partner providing the nonwoven materials, TFP was established nearly 30 years ago and is part of James Cropper plc, TFP is a wet-laid nonwoven manufacturer, producing

a diverse range of high-performance veils for an array of challenging applications. TFP's core capability is working in partnership with customers to provide solutions for technically demanding challenges.

The project found that the ORNL carbon tow (Figure 2) had a low tensile strength of 416 ksi and would need a new conversion process to chop the fiber material so it could be made into a nonwoven sheet using the wet laid manufacturing process (Figure 3). Due to this situation TFP was unable to convert and disperse the ORNL carbon tow material into a nonwoven veil with a uniform areal weight and pivoted to commercially available carbon fiber nonwovens. A commercially available industrial PAN carbon fiber was used to produce veils for the project.

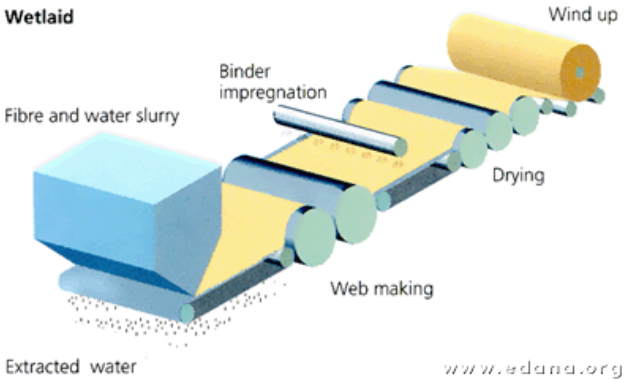


Figure 3 Wet Laid Generic Process

Figure 2: ORNL carbon tow

Another project discovery found that using a lower binder content of either Polyvinyl Alcohol (PVoH) or Carboxymethyl Cellulose (CMC) binders in carbon fiber substrates improves material solubility, and would reduce direct touch labor in part removal, but found the CBAM thermoplastic powder deposition causes residual thermoplastic material to remain throughout the printed carbon fiber sheet which has a negative impact removing the CBAM parts from the build block.

We recommend further work be conducted on reducing the level of residual thermoplastic powders remaining on the printed CBAM carbon fiber sheets to harvest the benefits of the soluble binder materials.

After resolving the CBAM residual powder matter, commercialization of a soluble binder carbon fiber veil will be driven by customer requirements and follow a standard product development and commercialization process.

3. INTRODUCTION

The CBAM process is a digital driven build process that manufactures complex thermoplastic composite parts directly from CAD files, eliminating the need for complex tooling, reducing skilled touch labor and simplifying supply chain logistics.

The conventional manufacturing process of producing composite parts is a very expensive process requiring a high degree of skilled touch labor, fabrication tooling therefore limiting the number of industrial use cases. The CBAM process eliminates tooling, reduces touch labor, and improves material recyclability which lowers composite part costs and opens up new composite market applications.

Potential CBAM light weight applications will include but are not limited to automotive components, aerospace and military products, electronics components and tooling, and oil and gas equipment.

According to U.S. Department of Energy Vehicle Technology office a 10% reduction in vehicle weight can result in a 6%-8% fuel economy improvement, since it takes less energy to accelerate a lighter object than a heavier one. Using lightweight composite components and high-efficiency engines enabled by advanced materials in one quarter of the U.S. fleet could save more than five billion gallons of fuel annually by 2030.

The commercialization plan will be driven by the demand of applications found in the automotive, UAV, air mobility and motor sports industries. Near term applications could include battery enclosure cases, fuel cell components, light weight structures and high temperature electronics surface mount tooling. The timing of product commercialization of a lower cost carbon nonwoven will depend on market demand and size. Future market estimate analysis will need to be conducted to determine when IO and the nonwoven material suppliers decide to bring forward this lower cost soluble binder nonwoven product offering.

4. BACKGROUND

The CBAM technology is a “state of the art” manufacturing solution with eleven issued patents and various invention disclosures and provisional patents pending. The CBAM technology is the only commercially available additive manufacturing technology today that uses nonwoven fiber substrates and thermoplastic powder matrix material.

The project goal was to identify a lower cost carbon fiber nonwoven substrate that would generate a lower final part cost. This can be accomplished by eliminating the touch labor in the CBAM part removal process step, expanding CBAM build capabilities to complex part geometries and allowing for the recovery of the unfused carbon fibers for recycling from part build block.

The plan was to evaluate different carbon materials including ORNL’s low-cost carbon tow made with lower cost hydrocarbon precursors, carbon fiber nonwovens sheet with different types of soluble binder types that can work in the CBAM process and yield composite parts with adequate material and dimensional properties.

The IACMI project team includes TFP a global nonwoven material provider, UAMMI manufacturing and composite expertise, Evonik Industries is a global specialty chemical company and IO the commercialization source of the CBAM technology. This IACMI team has the relevant technical expertise and product commercialization experience.

5. RESULTS AND DISCUSSION

5.1 Technical Approach

A key feature of the CBAM process is that the application of printed material to the substrate results in a change in the behavior of the substrate when subjected to the chosen method for removal of unprinted material. This allows unprinted materials to be removed from a build block without damaging the printed regions of the block. The current method for part removal is currently sandblasting, where the polymer powder (applied as part of the printing process), once melted and compressed, protect the fibers from the effects of the sandblast medium. Sandblasting works very well in the CBAM system but has some significant drawbacks including:

- The removed material cannot be easily recycled into material which could be used to make new substrates.
- The removal of large amounts of material can be time consuming, and while automation can reduce manual labor for this task, this process currently requires significant touch labor.

One possible method of removing the majority of fibers in a recyclable form with little or no manual labor, would be to dissolve the binder holding the fibers together in the substrate, allowing any fibers not actually embedded in the part to be 'washed' off the part in an automated removal bath. The objective of this work is to explore the possibility of using a soluble binder in the CBAM process to simplify the process and to increase the potential for recycling the fibers used in the process.

The primary goals of the program were, therefore:

- identify or create a suitable CBAM substrate material using carbon fibers and a soluble binder,
- verify that the material can be used in the CBAM process to manufacture additive parts,
- assess any change in mechanical properties compared to the current state of the art materials,
- understand any further improvements in materials or process required to allow the soluble binder to be used commercially.

The main phases of the project were:

- Production and evaluation of lab scale material samples
- Production of a larger scale trial run of materials
- Evaluation of the larger scale trial run materials
- Mechanical testing of baseline and trial materials

5.2 Lab scale samples of soluble binder hand sheets

A summary of the carbon fiber veils offered to IO for initial screening is shown in Table 1.

Table 1: Summary of small-scale samples

Product Reference	Sample Reference	Description
20301A (commercial grade)	20301A	17 g/m² Carbon veil with 10% PVOH binder
20301B (commercial grade)	B	17 g/m² Carbon veil with 10% PVOH binder
Hand sheet 1 (lab made)	A	17 g/m² Carbon veil with 3% PVOH binder
Hand sheet 2 (lab made)	C	17 g/m² Carbon veil with 5% PVOH binder
Hand sheet 3 (lab made)	D	17 g/m² Carbon veil with 10% PVOH binder
Hand sheet 4 (lab made)	F	17 g/m² Carbon veil with 10% PVOH binder
Hand sheet 5 (lab made)	G	17 g/m² Carbon veil with 10% CMC binder

Samples were either commercial grades and, therefore, small samples were available from the TFP marketing inventory, or were custom made for the project.

TFP produced veils using commercially available industrial PAN carbon fiber at two different fiber lengths (6mm & 12.5mm), two binder types (PVOH and CMC) and three different binder percentages (3%, 5%, 10%). The nonwoven product 20301A grade nonwoven had a fiber length of 6mm and 20301B product had a fiber length of 12.5mm.

TFP made the samples in the lab using a device called a hand sheet maker. This device replicates the formation process taking place on the pilot line and production machines whilst minimizing resource usage. The fibers are dispersed in an aqueous solution and filtered onto a metal screen or *'wire'* before being dried in an oven. Determination of the optimal processing conditions and drying can all be completed using this method (shown in Figure 4).

Figure 4 The hand sheet maker enables rapid evaluation of new nonwoven systems

The water solubility of the materials was tested by taking a 10 cm x 10 cm sample of each of the variants and immersing in 300 ml of cold water in a conical flask. Giving the flask a shake, the dispersibility of the material was determined by visual inspection.

IO Evaluation of lab scale samples

The samples were all regular and well-formed without any substantial delamination or surface roughness, though a little soft compared to the standard CBAM substrates.

The sheets were all tested with the solubility protocol described in Appendix A with the results that while all the samples broke down with sufficient agitation, sample B (20301B) performed the worst of these samples. Based on this result we checked the printability (as described in Appendix B) of the other samples and found that all samples performed similarly on powder uptake to IO's standard CBAM materials. Figure 5 shows the powder uptake in the printed areas for the hand sheet samples compared to a baseline for the standard CBAM substrate.

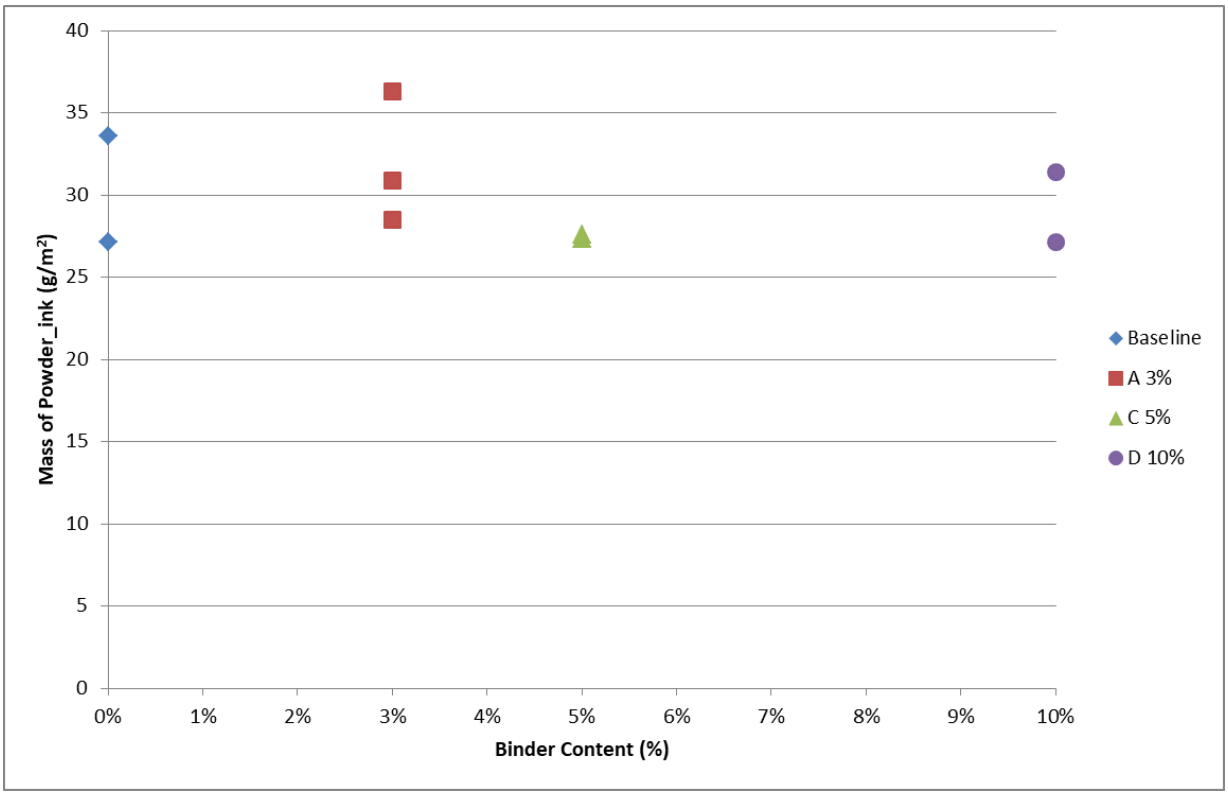


Figure 5 Powder Ink uptake on PVoH samples. (Note control sample shown at 0% binder)

Part Build

To test removability of the veils in a part we developed a small test piece than can be used to make a 1mm thick part requiring 20 nonwoven fiber sheets to build. Figure 6 shows the part design and the printed part. The sample part has a triangular cut out to assess the degree of bridging of fibers across gaps, and a series of holes of varying sizes to assess whether small holes can be cleaned out. In Figure 6 we can see the baked part (Right picture).

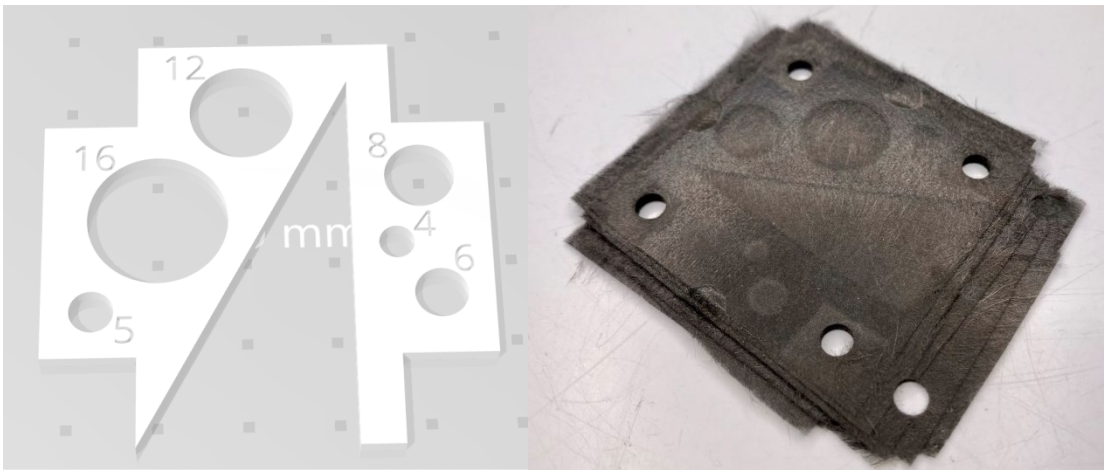


Figure 6 Sample Part design and baked part

.

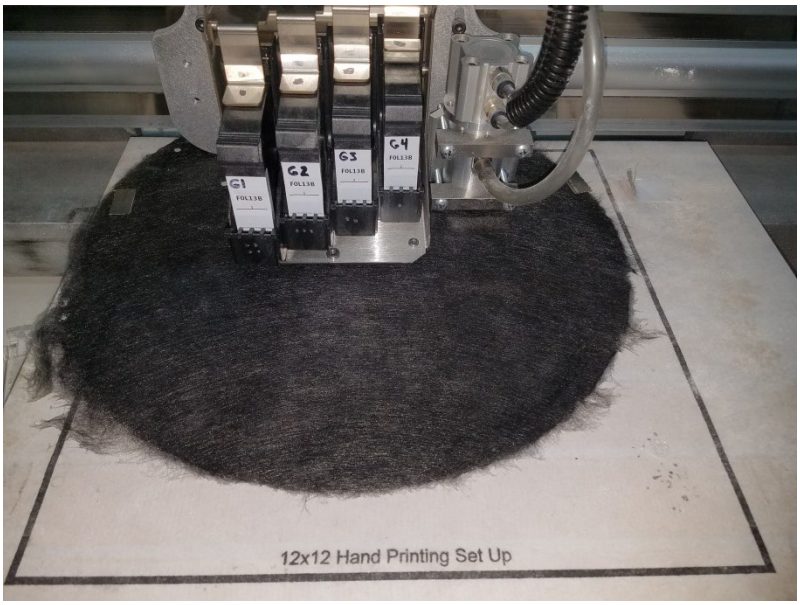


Figure 7 Printing on hand sheet sample

The most promising sample was the 5% low molecular weight material (C). A test part was made using Evonik Vestosint 1115 PA12 powder and baked following the standard CBAM part consolidation routine. Figure 7 shows the printing in progress on a sample veil.
To assess dissolution the part was soaked in water at 80ºC. Unfortunately, this did not result in the expected breakdown of the binder system, and while the unprinted areas softened a little this was not sufficient to significantly aid removal of the unprinted fibers.

We considered two possible causes for this:

- The PVoH had been stabilized during the heat treatment process and was less soluble as a result, or
- The residual powder in the unprinted areas stabilized the mat to an extent that prevented easy removal.

Based on preliminary testing it appeared that BOTH of these were responsible to some degree.

Revised Binder Options, (Carboxy Methyl Cellulose)

Two new sets of hand sheets were prepared by TFP, one set with the low molecular weight PVoH, and a second with an experimental Carboy Methyl Cellulose (CMC) binder (Samples E and F in Table 1). We carried out dissolution tests with the material for both as received and 'baked' sheets. It was clear that as previously seen the PVoH sheets were stabilized to some degree by the baking, and that the CMC dissolved very easily in hot water even after heat treating the sheets.

In the simple dissolution test we observed that the CMC binder sheets separated completely in hot water, once again the PVoH sheets softened but did not totally 'dissolve'.

As a final test IO manufactured a small (25mm x 25mm x1mm approx.) test sample using a manual CBAM printing system. The sample was made successfully and an attempt was made to dissolve the binder to release the excess fibers, this test was less successful, but sufficiently promising that we wanted to run some larger scale tests. For the larger scale tests, we needed a production trial quantity of material from TFP.

5.3 Trial material run

IO completed screening of the small samples and concluded that the carbon veil with 10% CMC binder showed the most promising results. A Pilot Line trial to manufacture this variant (product reference R2057-00) was then carried out by TFP. During the trial the veil architecture was formed, the binder was applied, and then the nonwoven was dried and reeled up. The TFP Pilot Line is shown in Figure 8. The trial was successful, and two rolls of 500mm (width) x 200m (length) were prepared. The material was characterized for areal weight (basis weight), thickness, binder content, tensile strength and water dispersibility, and the results for the two rolls are shown in Table 2.

Figure 8 TFP's Pilot Line facility

Table 2 Product results for R2057-00

Variable	Units	Result (Roll 1)	Result (Roll 2)
Grammage	g/m²	16.4	16.4
Thickness @ 10kPa	mm	0.199	0.195
Tensile MD	N/15mm	7.97	8.30
Tensile CD	N/15mm	3.57	3.70
Flash LOI	%	10.1	7.0
Disintegration in water	Y/N	Yes	Yes

5.4 CBAM builds with CMC Binder Fabric

Veil Transport through CBAM system

We received the rolls of production trial material. The first task was to sheet the material to our standard 12"x8" and 12"x12" sheets. At IO we use a sheeter slitter with drag knife slitters, and a guillotine sheeter blade. We found that the material was less stiff than our standard material which uses a polyester based binder, and that as a result the material did not slit cleanly, instead tearing on occasion. As a result, we cut the material to final size with die cutters. For production purposes we could improve this process by using an alternative slitting method (probably rotary blades).

We also tested the material on automated CBAM machines; we found a slightly higher rate of page transport jams than with the standard sheets, which is thought to be related to the sheet stiffness, which is noticeably lower than the standard materials. The overall sheets properties for feeding through the CBAM machine were not as good as the standard material, however not to a degree than cannot be countered with machine modifications if required.

PA12 tests, and issues

Our initial builds were simple parts to test binder solubility, using Evonik 1115 PA12 powders. To evaluate the solubility, we used small sections of the unprinted area of the build block. We started with hot water (80°C) but found that this did not allow the build block sections to soften or separate. To evaluate further we tested the materials with dilute acids and alkalis, and with significant agitation of the build block sections. These trials did not produce any improvement in performance.

The solubility test found a high level of residual of PA12 thermoset powder was getting embedded into the surface unprinted sheets of the carbon fiber test sheets and was sufficient enough to hold the test build block together even after the CMC binder was removed in the hot water bath of 80°C and hotter.

Microscopic examination of the printed sheets and measurements of the mass of residual powder tended to confirm this conclusion.

Residual Powder Reduction

The next set of tests were trials to reduce the residual powder in the unprinted areas while maintaining sufficient powder in the printed regions to form an acceptable part. We started with the Evonik 1115 PA12 by making modifications to machine settings. Unfortunately, we were not able to produce an acceptable print test with the 1115. Although the printing performance of this powder is good for our standard sandblasted materials, there is too much residual powder with our current machines to allow for easy removal of the excess fiber through dissolution of the binder.

Alternative Powders

We followed these tests up with tests on alternative powders that we had available, screening some of the trial powders that Evonik has provided to IO. Following these tests, we selected the Evonik powder with the lowest residual powder in unprinted areas as the most likely to produce an acceptable result. The best powder for this proved to be grade D390.

We made an initial test part with the D390 powder and investigated the behavior on immersing in 80°C water. We found that the material softened noticeably after ~5 minutes immersion, and although the unprinted areas did not come apart in the way unprinted sheets did it was possible to pull sections of the build away from the main block as either the PVoH or CMC binder in the nonwoven material softened.

Based on this set of tests it seemed unlikely for us to be able to achieve the main aim of the project without making changes in the manufacturing equipment or the materials beyond the scope of this investigation. As a result, we chose to investigate the degree of softening we had achieved with the materials and trials to date. We therefore made additional builds for mechanical testing, ‘pull-apart’ testing, and a small part.

5.5 Mechanical Testing of Materials

Pull apart testing

Since the residual powder prevented the desired breakdown of the unprinted areas to the desired degree, we investigated how much the soluble binder allowed the parts to be removed from the block without breaking. To investigate this further we designed a 'pull-apart' specimen comprising two printed areas 20mmx25mm to act as tabs separated by an unprinted area as a gage section.

To investigate the impact of too much residual thermoplastic powder remaining on the unprinted region of pull-apart specimen and created this test procedure to remove the final part in a dry and wet environment. Figure 9 shows a schematic of the pull-apart test specimen.

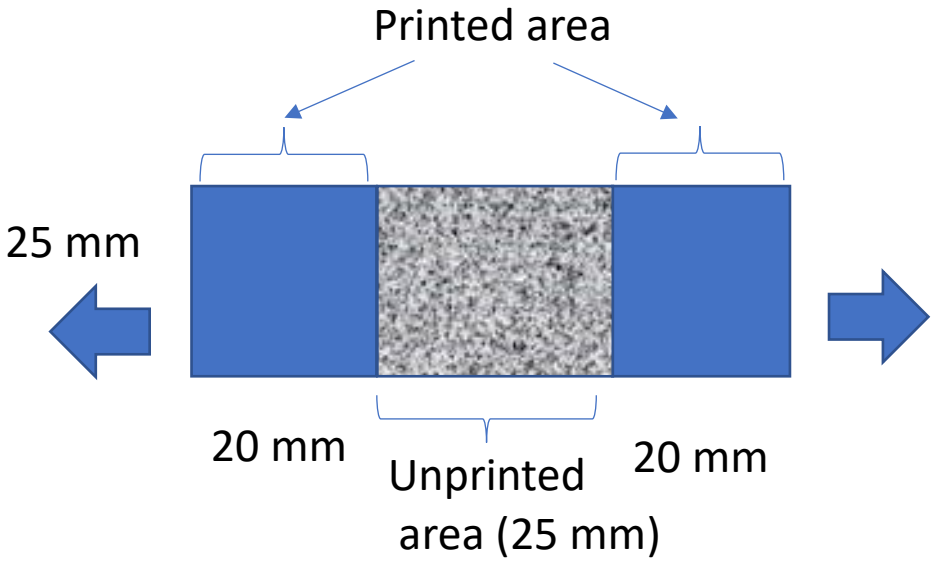


Figure 9 - Pull-Apart Test Specimen

The test pieces were cut from the completed build block with a sharp knife, and then tested in a tensile test machine in both dry and wet form. The 'wet' parts were soaked in 80ºC water for 45-60 minutes to ensure that any CMC binder that could be dissolved was fully removed.

Figure 10 shows the maximum load on the specimen during testing for the various specimens. We anticipated that the maximum load would depend on the separation distance, with 12.5mm fibers we expected a significant change in behavior at around the fiber length, as below that length there are fibers bridging from one tab to the other, whereas above that length there should not be.

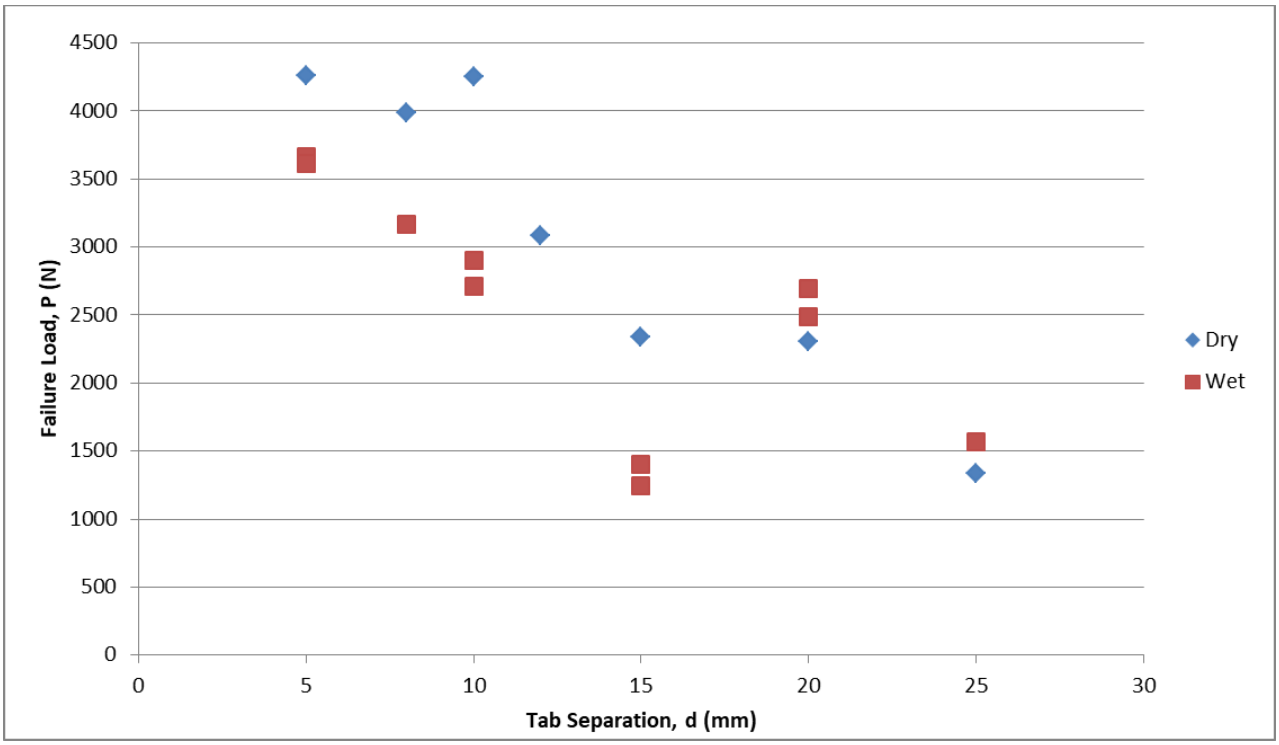


Figure 10 Pull Apart Load

The project found a shift in pull apart load behavior around 15 to 20mm tab separation distances for both specimens and found wet specimens pull apart load increases significantly.

For tab separation distances below 15mm, wet specimens had a lower failure load values in comparison to the dry test specimens.

We determined that the most likely cause of this behavior is that during testing the fibers in the parts with greater separation are more readily able to rotate than the specimens, and become more aligned with the test machine axis, as a result they carry more load than expected as they approach failure. In the case of the wet fibers the starkest difference is seen between 15mm and 20 mm separation where even though the separation increases, the load goes up instead of down.

Mechanical Testing

Mechanical testing in this project was primarily tensile testing to verify that the change in binder did not cause a loss of tensile strength in the final parts. Tensile (ASTM D638) and flexural (ASTM790) specimens were manufactured at IO using the standard CBAM veils with both PEEK and PA12 polymers and sent to University of Tennessee Composites Lab Knoxville for testing. Due to project time constraints, IO conducted in-house tensile testing of ten samples of the preferred CMC binder. A summary of the data is shown in Table 3. Details of the University of Tennessee testing are given in Appendix B. These are compared with the tensile tests completed on the soluble binder tests performed at Impossible Objects.

Table 3 Tensile Test Results

Binder	Polymer	Tensile Strength (MPa)	Tensile Modulus (GPa)	Strain to Failure (%))
Baseline	PEEK	155.7	13.1	1.2
Baseline	PA12	113.4	9.1	1.4
CMC Soluble	PA613	102.8	11.1	0.97

Based on these tests there is no significant loss of tensile strength to either of the soluble PVoH or CMC binder compared to the baseline, non-soluble polyester binder. Future work should be conducted to formulate the appropriate heating and pressing parameters to meet or exceed baseline material properties for the final part.

5.6 Manufacture of simple Part

As a final test we manufactured a sample part using the CMC binder carbon with the Evonik D390 powder. The actual part is a gasket for an engine component in an automotive application. The material printed well as can be seen in Figure 11.

Figure 11 Test part made with CMC binder carbon fabric and Evonik D390 PA613 powder

5.7 Conclusions

The commercially available carbon fiber substrates provided by TFP with the CMC binder produced good results for part solubility and show promising route to both easier fiber removal and recycling.

Further work is needed to the CBAM additive process reduce the residual powder in unprinted areas and enhance carbon fiber fabrics surface properties to improve sheet transport through the CBAM printer. There is no evidence that the carbon fiber CMC substrates produced similar mechanical properties to the current polyester veils used in the CBAM process.

6. BENEFITS ASSESSMENT

The U.S. Department of Energy calculates if the body structure of a car is made 30% lighter using carbon fiber, 50 tons of CO2 will be reduced per 1 ton of carbon fiber over a life cycle of 10 years; when the fuselage structure of aircraft is made 20% lighter using carbon fiber, on the other hand, 1400 tons of CO2 will be reduced under the same condition.

The project found carbon fiber nonwoven materials containing a soluble binder enables additive manufacturing of composite parts, eliminates part tooling, reduces build labor to remove final parts, improves material recycling and overall product sustainability.

7. COMMERCIALIZATION

Future product commercialization of the soluble carbon fiber nonwoven materials will be driven by market size and demand for a new CBAM material product offering. Light weight part applications will be the main factor that drives material commercialization and translate into millions of dollars of revenue for all ecosystem stakeholders including IO, nonwoven material suppliers and CBAM part suppliers.

8. ACCOMPLISHMENTS

No awards, publications or patents resulted from this short-term IACMI project. It is expected that future development work would likely yield patent filings, publications and industry awards.

9. CONCLUSIONS

The project found that CMC soluble binder is a viable material option for the CBAM process provided solutions can be found for eliminating the residual thermoplastic powder on the printed sheets that inhibits build block solubility and final part removal.

10. RECOMMENDATIONS

The CBAM printing technology will require further technology process development to reduce the amount of residual powder remaining on printed nonwoven sheet along with work on ultrasonic tank options and removal detergents. After solutions are found for these technical items the product commercialization of low-cost carbon nonwoven materials would depend on market pull and size.

11. BIBLIOGRAPHY

Composite and carbon fiber remanufacturing report. Guide to reclaimed carbon fiber & market applications.

https://www.adherent-tech.com/

Department of Energy, Clean Energy Manufacturing Innovation Institute for Composite Materials and Structures. Funding Opportunity Announcement (FOA) Number DE-FOA-0000977. DOE/EERE. 2014.
http://www1.eere.energy.gov/manufacturing/financial/solicitations_detail.asp?sol_id=760

https://www.energy.gov/eere/vehicles/lightweight-and-propulsion-materials

Heil JP, Cuomo JJ. Study and analysis of carbon fiber recycling, Master Thesis, North Carolina State University, Raleigh NC, USA: 2011.

Illing-Gunther H, Hoffman M, Gulich B. Nonwovens made of recycled carbon fibre by compounding with thermoplastic polymers. In: Proceedings of the 7th international CFK-Valley Stade convention "Latest Innovations in CFRP Technology" 2013

Piner-Hernanz R. Dodds C. Hyde J. Garcia-Serna J. Poliakoff M Lester E, Chemical recycling of carbon fibre reinforced composites in near critical and supercritical water. Composites Part A 2008; 39:454-61

Roux M, Dransfeld C, Eguemann N, Giger L, Processing and recycling of a thermoplastic composite fibre/PEEK aerospace part. In: Proceedings of the 16th European conference on composite materials (ECCM 16), 22-26 June 2014, Seville, Spain

12. APPENDICES

Appendix A – Dissolution and Printability Testing

12.1 Hot water binder dissolution testing

Samples

Samples should be cut from either sheets or build blocks. The size and shape of the sample should be recorded on the test report. For single sheet tests samples should be approximately 3cmx3cm.

Equipment

- Stainless Steel water bath
- Lab hot plate with magnetic stirrer
- Steel or Glass rod for stirring.

Method

Heat 500-1000ml of deionized water, (or other fluid) on the lab hot plate while stirring until it reaches 80°C. Add a sample of test material, and allow to soak for 30seconds, record any observations. After 30 seconds agitate the sample with the metal or glass rod, note if the sample retains its shape or starts to show signs that the binder is no longer holding the fibers together. Leave the sample in the water for a further 30 minutes. Repeat the agitation and record any changes in the sample.

12.2 CBAM Printability Testing

Printing test

Prepare 6 12”x8” sheets of CBAM substrate.
Weigh each sheet on a scale reading to +/- 0.001g.
Start a printer and check it is operating normally. Record the air knife pressure, star wheel vacuum level and conveyor speed.
Pass three sheets through the powdering section without printing. (Allow at least 10 seconds between sheets). Collect the sheets from the outfeed conveyor and reweigh and record the mass. Calculate the residual powder by dividing the mass difference by the fiber area.
For the remaining 3 sheets print a rectangle of known size (either 4”x4”, or another standard size available on the printer.) Collect the sheets from the outfeed conveyor and reweigh and record the mass. Calculate the ink+ powder load by dividing the mass difference by the printed area. Typically for production use this value is acceptable, if the ink and powder need to be recorded separately either apply a standard ink allowance ($1g/m^2$ for standard CBAM fluid) or measure the ink deposition rate by printing without powder.

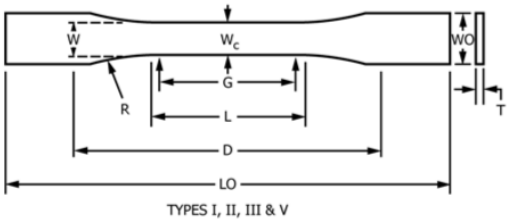
Appendix B – Mechanical Test Results

12.3 Static Tension and Flexure Characterization

Dayakar Penumadu, PI ;Joey Michaud (Undergraduate) ;Stephen Young (Post Doctoral Research Associate)
February 28, 2020

Tensile Testing

Tensile Samples (ASTM D638)



TYPES I, II, III & V

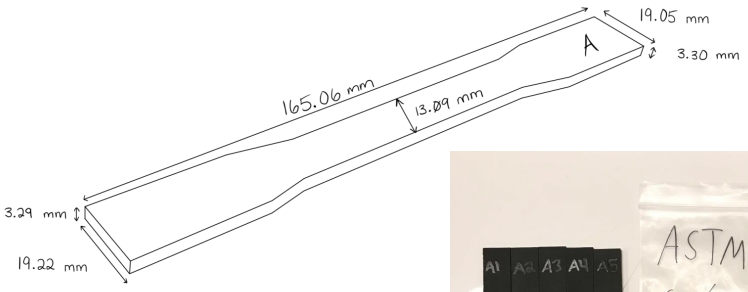
Dimensions (see drawings)	7 (0.28) or under		Tolerances
	Type I	Type II	
W—Width of narrow section[E,F]	13 (0.50)	6 (0.25)	±0.5 (±0.02)[B,C]
L—Length of narrow section	57 (2.25)	57 (2.25)	±0.5 (±0.02)[C]
WO—Width overall, min[G]	19 (0.75)	19 (0.75)	+ 6.4 (+ 0.25)
WO—Width overall, min[G]	...	...	+ 3.18 (+ 0.125)
LO—Length overall, min[H]	165 (6.5)	183 (7.2)	no max (no max)
G—Gage length[I]	50 (2.00)	50 (2.00)	±0.25 (±0.010)[C]
G—Gage length[I]	...	...	±0.13 (±0.005)
D—Distance between grips	115 (4.5)	135 (5.3)	±5 (±0.2)
R—Radius of fillet	76 (3.00)	76 (3.00)	±1 (±0.04)[C]
RO—Outer radius (Type IV)	...	...	±1 (±0.04)

ASTM D638: Standard Test Method for Tensile Properties of Plastics

Crosshead rate: 5 mm/minute for modulus determinations

THE UNIVERSITY OF TENNESSEE KNOXVILLE

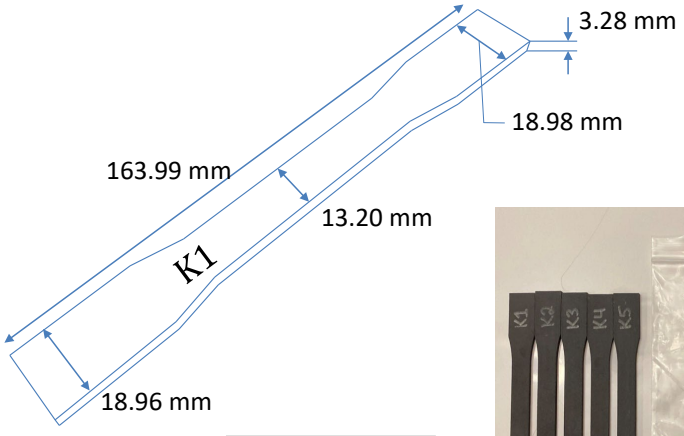
Nylon-12 Tensile Samples, Dimensions and Samples Photograph



N = 10
10 samples tested

THE UNIVERSITY OF TENNESSEE KNOXVILLE

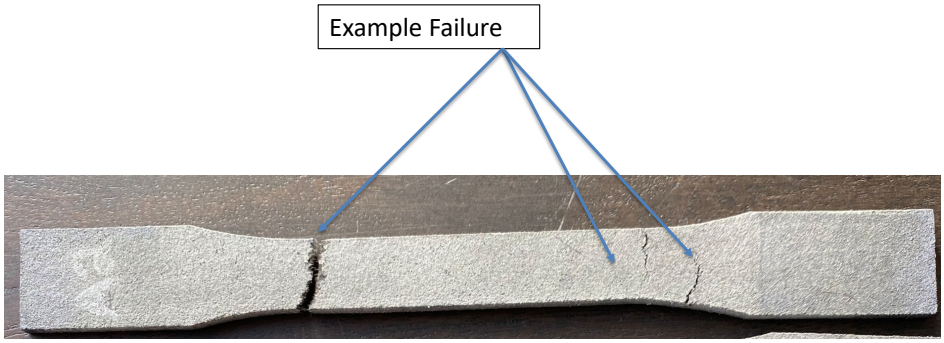
PEEK Tensile Samples, Dimensions and Samples Photograph



N = 10
9 samples tested

THE UNIVERSITY OF TENNESSEE KNOXVILLE

Nylon 12 Tensile Sample after mechanical failure

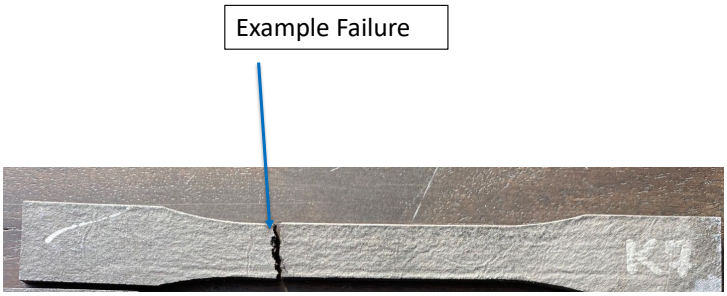


Sample A2

5 kip MTS
Based on ASTM D638
Crosshead rate: 5 mm/minute

THE UNIVERSITY OF TENNESSEE KNOXVILLE

PEEK Tensile Sample after mechanical failure

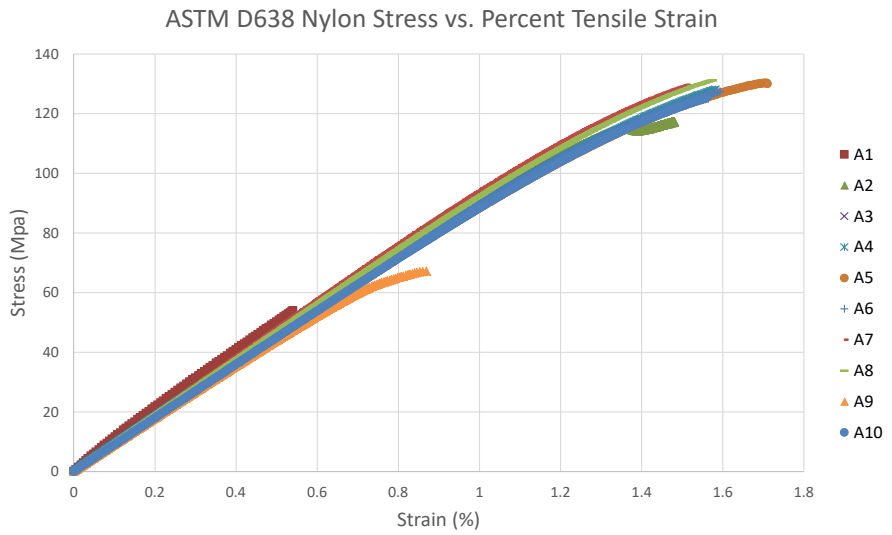


Sample K7

THE UNIVERSITY OF TENNESSEE KNOXVILLE

Tensile Stress/Strain Behavior

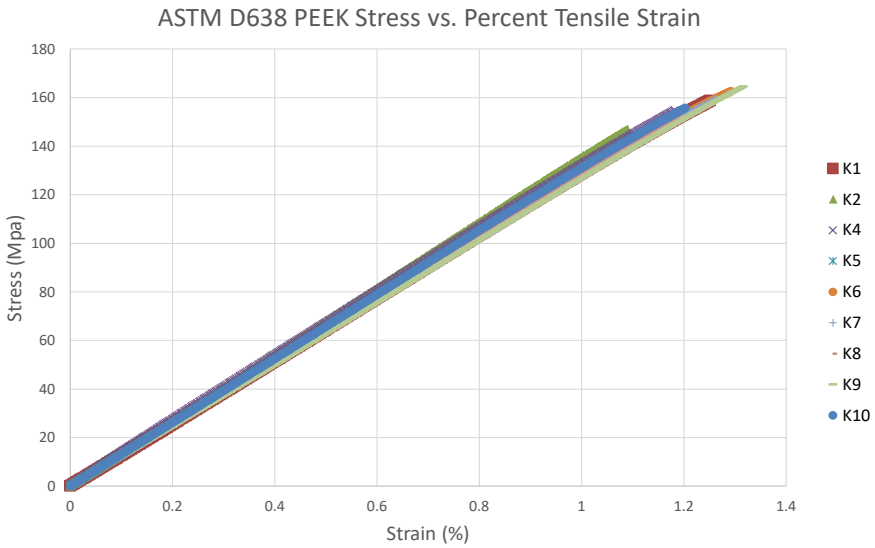
Nylon 12:



THE UNIVERSITY OF TENNESSEE KNOXVILLE

Tensile Stress/Strain Behavior

PEEK:



THE UNIVERSITY OF TENNESSEE KNOXVILLE

Tensile Mechanical Properties for Nylon Samples

Sample ID	Tensile Modulus* (GPa)	Tensile Strength (MPa)	Eng. Failure Strain (%)
A1	10	54	0.54
A2	9	116	1.37
A3	9	125	1.56
A4	9	128	1.58
A5	9	130	1.70
A6	9	127	1.59
A7	10	130	1.51
A8	9	131	1.57
A9	9	67	0.87
A10	9	126	1.56
Mean	9.1	113.4	1.39
Std Dev	0.317	28.334	0.376
Max	10	131	1.70
Min	9	54	0.54
CV (%)	3.47	24.99	27.16

* Chord modulus calculated at 1000 and 7000 με

THE UNIVERSITY OF TENNESSEE KNOXVILLE

Tensile Mechanical Properties for PEEK Samples

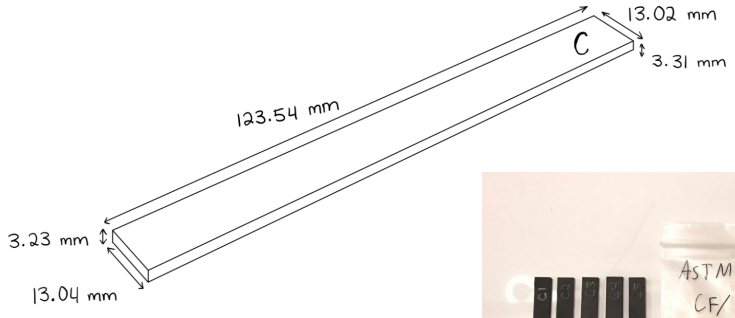
Sample ID	Tensile Modulus (GPa)	Tensile Strength (MPa)	Eng. Failure Strain (%)
K1	13	159	1.25
K2	14	147	1.09
K4	13	155	1.18
K5	13	147	1.14
K6	13	163	1.29
K7	13	159	1.26
K8	13	152	1.19
K9	13	165	1.32
K10	13	156	1.20
Mean	13.1	155.7	1.21
Std Dev	0.252	6.352	0.073
Max	14	165	1.32
Min	13	147	1.09
CV (%)	1.93	4.08	5.99

* Chord modulus calculated at 1000 and 7000 με
- Sample K3 not included

THE UNIVERSITY OF TENNESSEE KNOXVILLE

Flexure Data

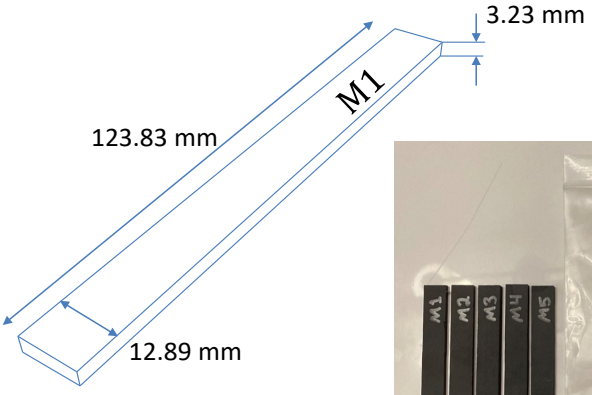
Nylon-12 Flexural Samples, Dimensions and Samples Photograph



N = 11
11 samples tested

THE UNIVERSITY OF TENNESSEE KNOXVILLE

PEEK Flexural Samples, Dimensions and Samples Photograph

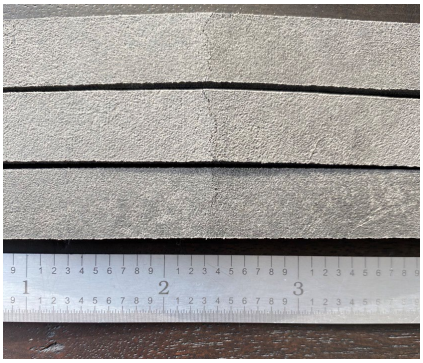


N = 11
11 samples tested

THE UNIVERSITY OF TENNESSEE KNOXVILLE

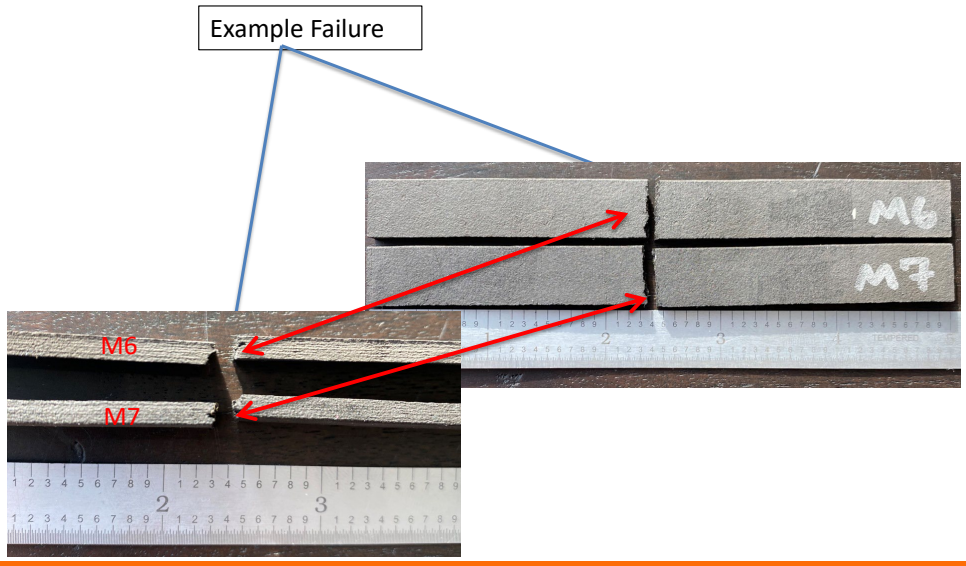
Nylon 12 Flexural Samples after mechanical failure

Example Failure



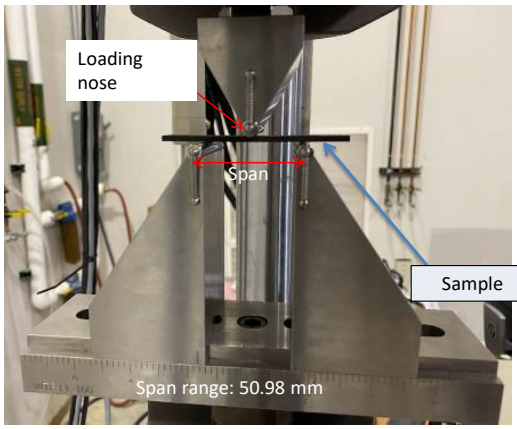
THE UNIVERSITY OF TENNESSEE KNOXVILLE

PEEK Flexural Samples after mechanical failure

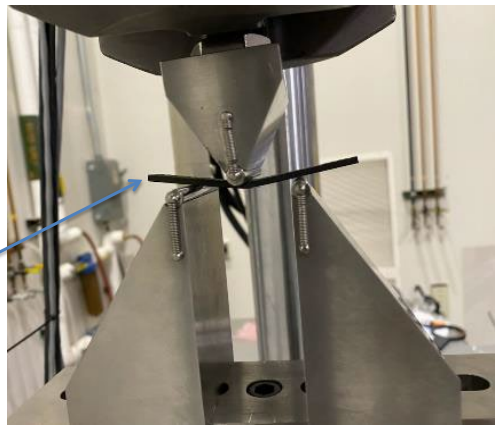


THE UNIVERSITY OF TENNESSEE KNOXVILLE

Flexural Mechanical Testing Setup



Before Loading



After Mechanical Loading*

Load Frame: MTS Mode 853
Load Cell: 5 kip (25kN), Model 662.204-05
Based on ASTM D790

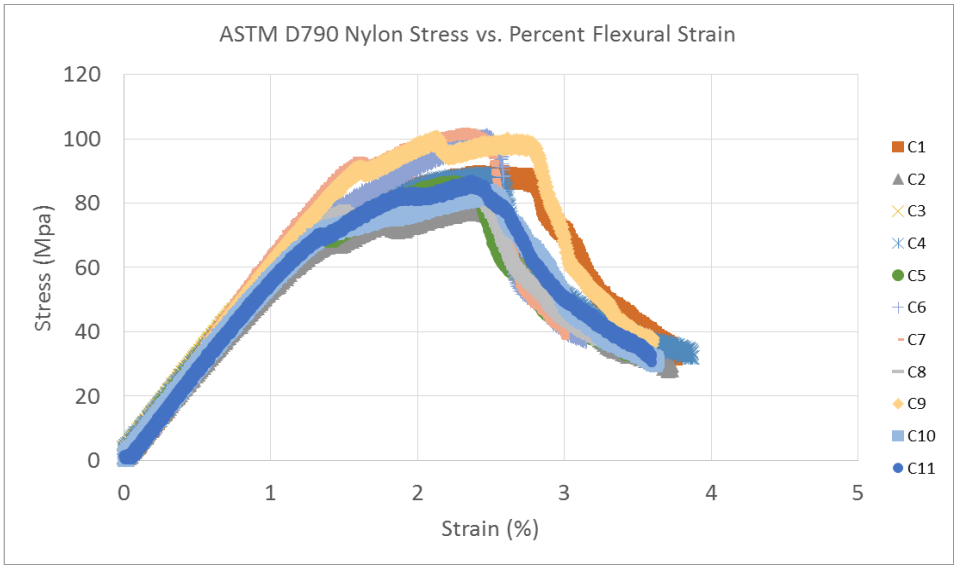
Samples tested n = 22:
11 Nylon samples, 11 PEEK samples

*Note: Due to their more brittle nature, PEEK samples did not stay in place, and fell apart instead

THE UNIVERSITY OF TENNESSEE KNOXVILLE

Flexural Stress/Strain Behavior

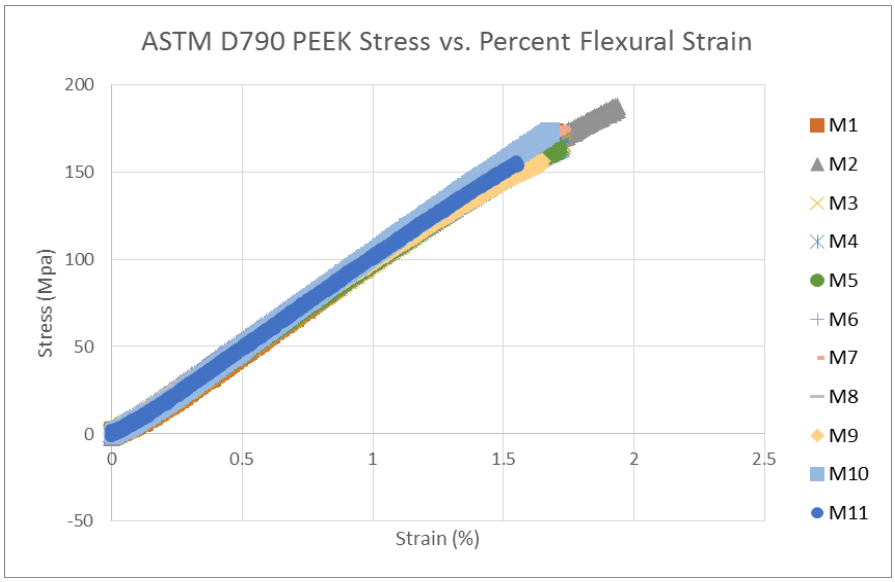
Nylon 12:



THE UNIVERSITY OF TENNESSEE KNOXVILLE

Flexural Stress/Strain Behavior

PEEK:



THE UNIVERSITY OF TENNESSEE KNOXVILLE

Flexural Mechanical Properties for Nylon Samples

Sample ID	Nominal Length (mm)	Width (mm)	Thickness (mm)	Span (mm)	Modulus (GPa)	Failure Stress (MPa)	Failure Strain (%)
C1	123.5	12.94	3.21	51.36	6	90	2.53
C2	123.4	12.75	3.20	51.25	5	79	2.32
C3	123.0	12.99	3.20	51.20	6	86	2.33
C4	123.3	12.74	3.19	51.08	6	89	2.44
C5	123.4	12.95	3.18	50.95	6	87	2.26
C6	123.6	12.71	3.13	50.09	6	101	2.47
C7	123.7	12.65	3.20	51.27	7	103	2.29
C8	123.3	12.81	3.19	51.02	6	86	2.35
C9	123.5	12.81	3.17	50.77	6	101	2.13
C10	123.1	12.75	3.18	50.87	6	84	2.46
C11	123.2	12.93	3.19	50.96	6	87	2.37
Mean	123.4	12.82	3.19	50.98	5.9	90.4	2.36
Std Dev	0.211	0.11	0.02	0.35	0.311	7.807	0.112
Max	123.7	12.99	3.21	51.36	7	103	2.53
Min	123.0	12.65	3.13	50.09	5	79	2.13
CV (%)	0.17	0.89	0.68	0.68	5.23	8.64	4.74

*Modulus: based on strain range of 0.1 – 0.7%

THE UNIVERSITY OF TENNESSEE KNOXVILLE

Flexural Mechanical Properties for PEEK Samples

Sample ID	Nominal Length (mm)	Width (mm)	Thickness (mm)	Span (mm)	Modulus (GPa)	Failure Stress (MPa)	Failure Strain (%)
M1	123.5	12.85	3.16	50.63	11	179	1.69
M2	123.3	12.91	3.12	49.89	10	188	1.94
M3	123.4	12.74	3.15	50.41	10	167	1.73
M4	123.5	12.87	3.17	50.79	10	165	1.72
M5	124.1	12.81	3.20	51.14	10	164	1.71
M6	123.4	12.85	3.15	50.36	11	170	1.68
M7	123.5	12.81	3.17	50.68	11	176	1.72
M8	123.6	12.85	3.16	50.49	10	160	1.60
M9	124.2	12.84	3.18	50.85	10	156	1.65
M10	123.5	12.79	3.23	51.60	11	174	1.68
M11	123.7	12.78	3.18	50.95	10	155	1.63
Mean	123.6	12.83	3.17	50.71	10.4	168.6	1.70
Std Dev	0.288	0.05	0.03	0.45	0.232	10.063	0.088
Max	124.2	12.91	3.23	51.60	11	188	1.94
Min	123.3	12.74	3.12	49.89	10	155	1.60
CV (%)	0.23	0.37	0.88	0.88	2.24	5.97	5.19

*Modulus: based on strain range of 0.1 – 0.7%

THE UNIVERSITY OF TENNESSEE KNOXVILLE