Material Characterization of a High Performance Additive Manufacturing Material for Efficient Component Design

Dr. Fayyaz Rehman[1], John Diston[2]

[1]*Associate Professor, Warsash School of Maritime Science and Engineering, Solent University, Southampton, UK*
[2]*Manufacturing Engineer, Atomic Weapons Establishment (AWE), UK*

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Abstract - *Additive manufacturing (AM) or 3D printing, is seen amongst some technologists, designers and engineers as the technology of the future. This is because of the advantages AM can bring to the design and development of creative and innovative products. The dream of AM one day replacing conventional manufacturing techniques may slowly become a reality. However there are many challenges to overcome before then, these include machine and material costs, production speed, attitudes towards new, often expensive AM technologies and a lack of reliable material design data. This paper will outline a solution to the lack of available material design data through a material characterization regime that will enable efficient component design, also enhancing how AM is perceived and promoting its adoption as a viable manufacturing technique. The development of the material characterization regime focuses on ULTEM 9085, determining which material properties were required, ensuring efficient dimensional measurement and test methods were used, before the manufacture and testing of tensile and compressive samples.*

Key Words: Additive manufacturing (AM), 3D printing, material characterization, finite element analysis (FEA)

1. INTRODUCTION

The inspiration for this project originated from a fairly specific problem encountered at the authors' place of work. A relatively large aerospace component containing a relatively small internal structure, manufactured from ULTEM 9085 on a Fortus 400mc, additive manufacturing (AM) machine needed to be simulated to find out how it would behave in tension and compression.

Stratasys [1] describes ULTEM 9085 [2] as a fused deposition modelling (FDM) thermoplastic material considered ideal for high performance, high temperature applications because of its fire, smoke & toxicity (FST) retardant materials rating and high strength-to-weight ratio. It empowers the design and manufacture of advanced functional prototypes and production parts ideal to be used in aerospace industry. ULTEM 9085 is a desirable engineering material and to take full advantage of the benefits it affords, the material requires characterising.

Any meaningful simulation of AM designed parts through FEA software requires basic material properties, for example, Young's modulus, ultimate tensile strength (UTS) and density. This research highlighted how important material properties are to the design process since FEA is a tool used to predict how parts will perform in service before testing can be done.

Although material property data for ULTEM 9085 is given by various suppliers on their websites, however that data is gathered through specific material testing standards through printing specimens using their own specific 3D printing machines. Also material's physical properties vary from supplier to supplier. In order to print aerospace production ready components with the author's 3D printing machines, reliable data was not readily available as very high variation in properties from different sources can be seen, therefore an investigation into how to successfully characterise AM materials using ULTEM 9085 as a starting point was required.

The investigation needed to result in a material characterization solution that provides material data and a guide for engineers looking to understand new and existing AM materials for safety critical, end use parts. This paper presents the methodology of research which includes predictions for a simulation that was validated via mechanical testing and the evaluation of material properties which are used in the characterization of ULTEM 9085 polymer and subsequently the creation of material specifications, for the use of 3D printing production ready aerospace components

2. MATERIAL CHARACTERIZATION

Material Characterization refers to the broad and general process by which a material's structure and properties are probed and measured. The scope of the term often differs; some definitions limit the term's use to techniques which study the microscopic structure and properties of materials while others use the term to refer to any materials analysis process including macroscopic techniques such as mechanical testing, thermal analysis and density calculation.

Additive manufacturing (AM) is being used for a variety of applications in industry. It is noted that not all applications of AM require material characterization as shown in table 1. AM technologies resulting in the manufacturing of prototypes/form/fit models, toolings and fixtures and end use components without quality requirements do not require detailed material characterization.

Table -1: AM material characterization decision matrix

Intended use of AM technology	Perform material characterisation?
Prototypes/Fit, form, function models only	No
Tooling & fixtures	No
End use components without a quality requirement	No
Safety critical end use components with performance simulation	Yes
Safety critical end use topologically optimised parts	Yes
Research, development and training	Yes

Hallgrimsson [3] highlights how the intended purpose of the AM machine also needs understanding, if it was purchased solely with the creation of rapid prototypes in mind then the properties of the materials are not important. Similarly, tooling and fixtures simply need to work, they will be made then used, an understanding of how the material will perform in the intended environment is essential but once determined a comprehensive understanding of the material properties is unnecessary. As with tooling and fixtures, end use components that aren't required to fulfil any quality requirements apart from simply working in an intended environment don't warrant a complete understanding of the material properties. However as table 1 suggests, end use safety critical components and for research and development, material characterization is absolutely vital.

2.1 Why isn't AM material design data easily available?

AM machine manufacturers are in the machined selling business, they are likely to only provide the best results and since competition exists between manufacturers, it isn't difficult to imagine them wanting to keep detailed material testing results to themselves. Moreover each material supplier provides conflicting and varied values of same mechanical properties like ultimate tensile strength and tensile modulus of ULTEM 9085(Table 2), which makes it necessary to characterize AM material before using it for critical applications as mentioned in table 1.

Table -2: UTS and Tensile Modulus of ULTEM 9085 from various suppliers (Source: Senvol database [4])

Material Supplier	Ultimate Tensile Strength (MPa)	Tensile Modulus (MPa)
INTAMSYS	86	2230
SABIC	45-80	2176-2555
STRATASYS	42-69	2150-2270
ARGYLE/BOLSON MATERIALS	74	3440

The variation in UTS and tensile modulus of ULTEM 9085 sufficed the need for the authors to carry out material characterization of ULTEM 9085 before it being used in safety critical end use aerospace components.

2.2 Optimal material characterization with results validation process

An optimal material characterization procedure involves a material testing results validation process as well. Careful planning is required to devise the procedure of an optimal material characterization process. The process involves communication with various personnel of different departments. Based upon the experience of characterization of ULTEM 9085 at authors' work place, following generic procedure is recommended for the optimal material characterization involving the results validation process as well:

- Additively design and manufacture samples of a test component with the required AM process in mind.
- Normalize the specimens at the test lab temperature for 2 days according to American Society of Testing Materials (ASTM) standard.
- Decide which performance/design characteristic (for example how much tension/compression can be applied to the design) and which material properties need to be gathered (for example, full stress/strain curves, density and Young's Modulus, more maybe required) you need to know and perform mechanical testing.
- Perform statistical analysis (e.g. mean and standard deviation of results, more analysis maybe required).
- Perform simulation/analysis filling out the material property data with the results gathered from mechanical testing.
- Compare actual test results to expected simulation results.

If acceptable and if agreed, consider the results validated and use test results in future simulation and analysis of the design, however it is advised that this whole process be a continuous cycle, this will ensure reliability of results and give a current machine state as the material properties may degrade as the machine ages. It will also improve/reduce any difference between simulation and actual performance.

3. MATERIAL CHARACTERISATION PROCESS OF ULTEM 9085

The aforementioned procedure was used to characterize ULTEM 9085 AM material before it is being used for manufacturing end use safety critical components. Important steps of this process are explained below:

3.1 Design and Manufacture of Test Specimens

Using a CAD software, type 1 tensile test specimens were modelled according to ASTM D638-14 Standard Test Method for Tensile Properties of Plastics as shown in figure 1.

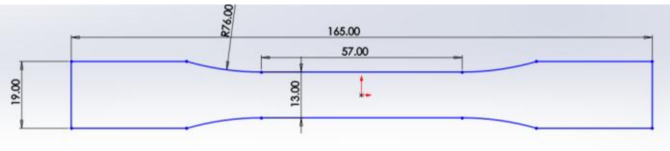


Figure 1: Type 1 specimen according to ASTM D638–14 Standard Test Method for Tensile Properties of Plastics 3.2 mm thick

Consideration to part orientation is required because, according to Stratasys' Technical Application Guide for FDM Jigs and Fixtures [5], how the part is orientated will effect part strength, surface finish and the amount of support used, hence for the design of experiment in this research 15 specimens were labelled with A1 to A15 along with corresponding three orientations (XYZ, XZY, YZX) as the part is built vertically upwards as shown in figure 2

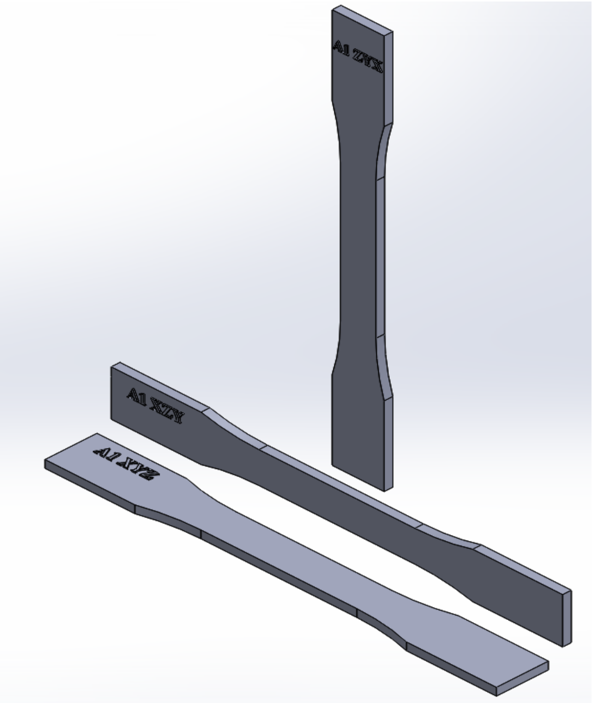


Figure 2: Three different build orientations (XYZ, XZY and YZX)

In addition to tensile specimens, 10 solid ULTEM 9085 round bars 50mm diameter and 50.4mm high (figure 3) are also produced as compression specimens. Both these two types of specimens were normalized at the test lab temperature for 2 days according to ASTM standard.

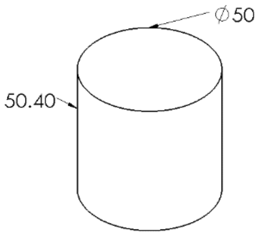


Figure 3: Compression Specimen

The AM machine being used was the Stratasys Fortus 400mc which uses Fused Deposition Modelling (FDM) Additive Manufacturing Technology.

3.2 Selection of Desired Material Properties for Investigation

Since ULTEM 9085 has to be used for end use safety critical aerospace components, so for an optimal design and additively manufactured ULTEM 9085 components, the essential properties which are investigated are Tensile Strength, Tensile Modulus, Tensile Elongation, Compression Modulus, Specific Gravity and Poisson's Ratio.

3.3 Mechanical Testing

The aim of the experiments were to provide material property data and show how important reliable material properties are when trying to predict component behavior. The first experiment was the testing of 45 ASTM D638 standard Type 1 'dog bones', 3.2mm thick, 15 were manufactured in each orientation (X,Y and Z) and pulled to failure using an Instron 5583 mechanical testing machine. The machine was fitted with a 100kN load cell, with the Poisson's ratio being measured using the extension of the gauge length between the grips, giving the axial strain and a transverse extensometer measuring the transverse strain. Of the 15 in each orientation, 5 were pulled with a strain rate of 5mm/min, then 5 at 10mm/min and finally 5 a 15mm/min. 15 more ASTM D638 standard type 1 'dog bones', 5mm thick, built in the XYZ orientation, were pulled to failure using the same machine, again 5 were pulled with a strain rate of 5mm/min, then 5 at 10mm/min and finally 5 at 15mm/min, to compare the dimensional proportionality of the materials properties.

The second experiment was a compression test on 10 solid ULTEM 9085 round bars 50mm diameter and 50.4mm high, predictions were made using a simulation package and simple calculations. The experiment involved applying a 30kN compressive force at 5mm/min using the same Instron 5583 machine and 100kN anvils and load cell.

A force threshold of 10N was used to automatically zero the compressive extension at the beginning of each test and the deflection at 10kN and 20kN and finally at 30kN was noted through analysis of the raw data results.

4. RESULTS OF TESTING

A range of different properties of ULTEM 9085 as mentioned in section 3.2 were calculated from the mechanical testing experiments results. These properties characterized the design requirements for the end use safety critical aerospace components manufactured at authors' workplace.

4.1 Tensile Properties

Stress strain curves for 3.2 mm specimen at three different strain rates are shown in figure4:

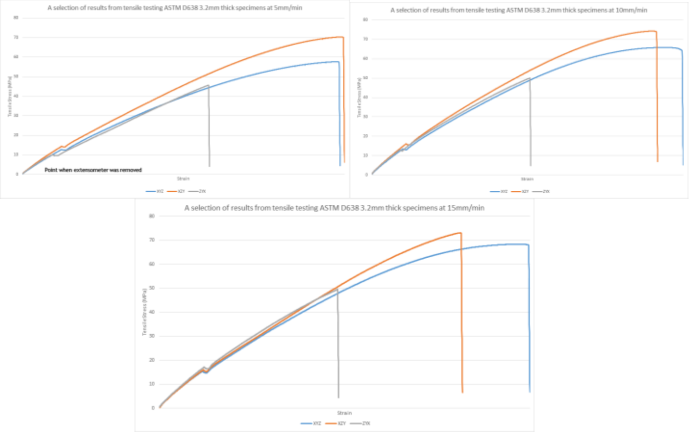


Figure 4: Stress/Strain Curves for 5, 10 and 15 mm/min strain rate for 3.2 mm thick specimen

Results in figure 5 show variation in the Young's modulus of the 3.2mm thick specimens in relation to strain rate and build orientation (figure 6).

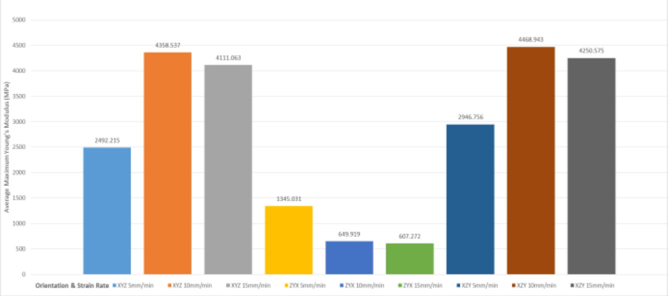


Figure 5: Mean Young's modulus of all 45 ASTM D638 3.2mm thick tensile specimens

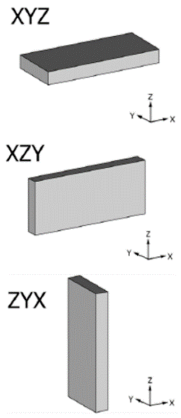


Figure 6: Specimen Orientation

Another set of XYZ oriented tensile test specimens 5mm thick were pulled. Table 1 show the mean Young's modulus results compared with the 3.2mm thick specimens. Most notable was how little difference the strain rate made to the results.

XYZ	Mean Young's modulus (MPa)			
Strain Rate → Thickness ↓	5mm/min	10mm/min	15mm/min	Mean Young's Modulus (MPa)
5mm	2284.028	2281.339	2185.402	2250.256
3.2mm	2492.215	4358.537	4111.062	3653.938

Table 1: Comparison of Mean Young Modulus of 3.2 mm and 5 mm thick specimens

4.2 Physical and Compression Properties

Figure 7 shows the probability distribution of the Poisson's ratio results and how little they deviate against strain rate and build orientation, although there was not enough data collected to be reliable for design, it does give the impression the materials Poisson's ratio is independent of strain rate and build orientation.

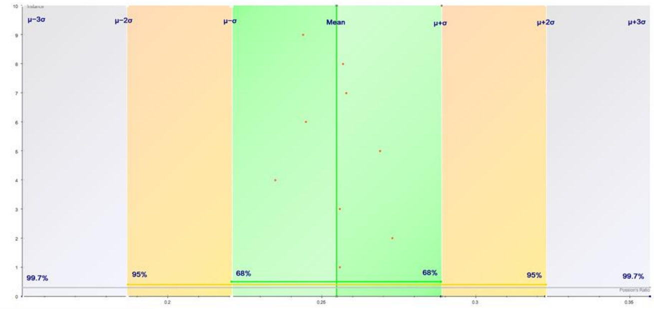


Figure 7: Distribution of mean Poisson's ratio

The deflection at 10kN and 20kN and finally at 30kN for 10 compression specimens was noted. Analytical calculations as well as finite element analysis (FEA) based ANSYS simulations (figure 8) were done to predict the deflection at 10kN, 20kN and 30 kN using the compression modulus value given in material selection database (Granti Mi) of PEI (Since ULTEM 9085 is a kind of generic Polyether Imide (PEI)) material.

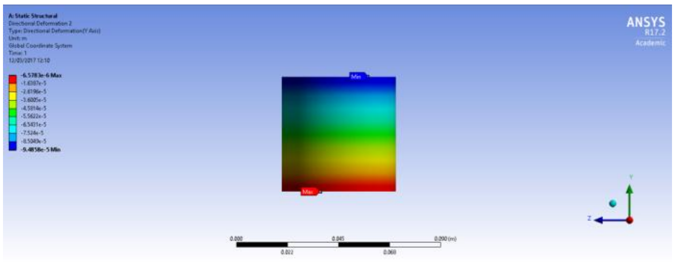


Figure 6: ANSYS based compression simulation results

Table-3: Predicted vs simulated and tested z-axis deflections and Young's modulus

Difference of predicted versus tested Z-axis deflection (mm)			
	Predicted	ANSYS	Tested
10kN	-0.0888	-0.0948	-0.5265
20kN	-0.1776	-0.1889	-0.7628
30kN	-0.2664	-0.2823	-0.9820
Young's modulus of solid PEI (MPa)	Granta Mi	ANSYS	Tested
	2890	2890	2492.21

15 density cubes of each side 10mm were manufactured at the same time as the 45 tensile specimens, the dimensions across pairs of faces of the cubes were taken in the approximate center of the cube and some increase towards the edges was noticed using the CMM machine. Mass of each cube was also measured. These measurements were used to calculate the mean specific gravity of the material to be 1.15 as shown in table 4.

Table-4: Results of density cubes measurements for specific gravity calculation

	Face 1	Face 2	Face 3	Volume (m^3)	Mass (g)	Mass (kg)	Density (kgm^3)	Specific gravity
Cube	mm	mm	mm					
1	10.37	10.13	10.02	1.05x10^{-6}	1.21	1.21x10^{-3}	1148.66	1.14
2	10.06	10.08	10.39	1.05x10^{-6}	1.2	1.20x10^{-3}	1138.16	1.13
3	10.02	10.02	10.43	1.04x10^{-6}	1.21	1.21x10^{-3}	1153.89	1.15
4	10.13	10.37	10.06	1.05x10^{-6}	1.22	1.22x10^{-3}	1153.20	1.15
5	10.03	10.41	10.05	1.05x10^{-6}	1.21	1.21x10^{-3}	1151.61	1.15
6	10.02	10.10	10.37	1.05x10^{-6}	1.21	1.21x10^{-3}	1151.26	1.15
7	10.07	10.01	10.39	1.04x10^{-6}	1.21	1.21x10^{-3}	1153.95	1.15
8	10.10	10.03	10.35	1.04x10^{-}	1.21	1.21x10^{-3}	1152.79	1.15
9	10.05	10.09	10.37	1.05x10^{-6}	1.21	1.21x10^{-3}	1149.65	1.14
10	10.39	9.98	10.02	1.04x10^{-6}	1.2	1.20x10^{-3}	1153.35	1.15
11	10.36	10.12	10.06	1.05x10^{-6}	1.2	1.20x10^{-3}	1136.85	1.13
12	10.05	10.37	10.08	1.05x10^{-6}	1.21	1.21x10^{-3}	1150.90	1.15
13	10.09	10.38	10.00	1.04x10^{-6}	1.23	1.23x10^{-3}	1173.24	1.17
14	10.11	10.05	10.42	1.05x10^{-6}	1.2	1.20x10^{-3}	1132.53	1.13
15	10.12	10.36	10.02	1.05x10^{-6}	1.21	1.21x10^{-3}	1149.97	1.14

5. CONCLUSIONS

There are two main challenges to AM technical benchmarking, the first is the development of new machines, every time a new machine is offered to the market, boasting an improved AM method, any planning in place to understand and benchmark it against either a rival or a conventional technology needs revising.

With no target in sight, these revisions appear endless and could prevent even the most determined of engineers from starting the process of benchmarking (it is not too difficult to imagine a kind of, "don't bother characterizing the material, there will be a new version of the machine out next year" mentality setting in amongst technologists).

This explains why FDM was chosen since it is probably the most mature and stable AM process and least likely to evolve significantly (the age of other technologies can be as little as a decade old and are still only fully understood by material scientists and the machines manufacturer) [6].

The second challenge is the relationship between rapid prototypes and end use parts, every time a poor quality prototype gets made with little to no thought to the process, it damages the perception of AM, this damage could prevent, or at least delay, the adoption of AM in the production of end use products [3].

Since this project was focused on how to gather good quality data, the surface has only been scratched with regards to how many results are required to yield reliable design data. The difference in the predicted and measured compressive deflections can be attributed to the manufacturing process having an effect on the properties of the material. The FDM process produces a material with a structure, rather than a homogenous bulk material.

The shape of the curves all seem fairly similar, they have behaved like a plastic, with only noticeable differences in length of the ZYX (vertical) orientated specimens. The XYZ orientated 5mm thick tensile specimens did not appear to be affected by the change in strain rate.

A significantly lower Young's modulus was measured for the ZYX (vertically) orientated tensile specimens proving the material is anisotropic. Taking an average of the whole set of results will yield incorrect material data because the large effect of build orientation, it would be an unfair comparison. The objective of this project was to show the importance of accurate material property data, while still in its infancy the population of results differs (table 5) from that quoted by the machine manufacturer (Stratasys). They do however caveat their material datasheets with a warning that owners of their machines should perform their own material characterization.

Table-5: Predicted vs simulated and tested z-axis deflections and Young's modulus

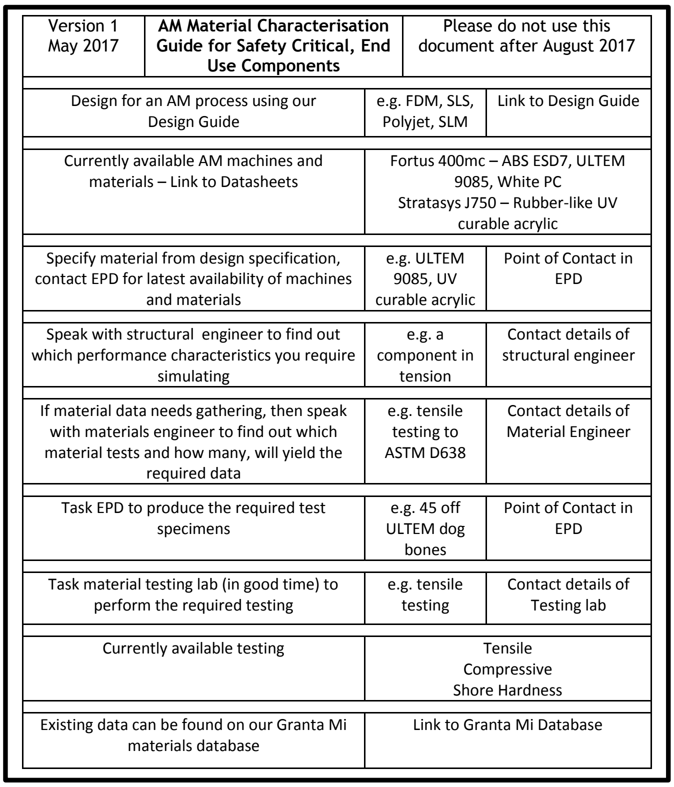
	Test Method	Stratasys Data Sheet		Test Method	Test result
Tensile Strength (Type 1, 0.125",0.2"/min)	ASTM D638	71.6 MPa	Tensile Strength (Type 1, 3.2mm, 5mm/min)	ASTM D638	59.95 Mpa
Tensile Modulus (Type 1, 0.125",0.2"/min)	ASTM D638	2200 MPa	Tensile Modulus (Type 1, 3.2mm, 5mm/min)	ASTM D638	2492.21 MPa
Tensile Elongation (Type 1, 0.125",0.2"/min)	ASTM D638	6%	Tensile Elongation (Type 1, 3.2mm, 5mm/min)	ASTM D638	7.57%
Compressive Strength	ASTM D695	104 MPa	Compressive Strength	-	-
Compression Modulus	ASTM D732	1930 MPa	Compression Modulus	In house test	1159.61 MPa
Specific Gravity	ASTM D792	1.34	Specific Gravity	In house test	1.15
Poisson's Ratio	-	-	Poisson's Ratio	In house test	0.254

4. RECOMMENDATIONS

A design driven approach to simulation and mechanical testing will avoid unnecessary testing, saving time and money. It will also inform everyone involved in the simulation, manufacture and testing of additively manufactured safety critical final use parts, why they are doing what they're doing, ensuring momentum is not lost and engineering projects get completed on time.

Material characterization could be planned and performed totally by an outside contractor but this could lead to not receiving value for money because you will not remain an intelligent customer. By having an understanding of the processes involved and the kind of results expected, sound decisions can be made based on internal knowledge and experience.

A guide (figure 7) was produced as a starting point for designers wanting to begin the characterization process. It should be kept up to date and reviewed regularly to ensure the advice and data is current. By being an informed, intelligent customer that uses design driven simulation and mechanical testing, with fully worked out requirements beforehand to perform meaningful material characterization, can avoid wasted time, money and effort.



Version 1 May 2017	**AM Material Characterisation Guide for Safety Critical, End Use Components**	Please do not use this document after August 2017
Design for an AM process using our Design Guide	e.g. FDM, SLS, Polyjet, SLM	Link to Design Guide
Currently available AM machines and materials – Link to Datasheets	Fortus 400mc – ABS ESD7, ULTEM 9085, White PC Stratasys J750 – Rubber-like UV curable acrylic	
Specify material from design specification, contact EPD for latest availability of machines and materials	e.g. ULTEM 9085, UV curable acrylic	Point of Contact in EPD
Speak with structural engineer to find out which performance characteristics you require simulating	e.g. a component in tension	Contact details of structural engineer
If material data needs gathering, then speak with materials engineer to find out which material tests and how many, will yield the required data	e.g. tensile testing to ASTM D638	Contact details of Material Engineer
Task EPD to produce the required test specimens	e.g. 45 off ULTEM dog bones	Point of Contact in EPD
Task material testing lab (in good time) to perform the required testing	e.g. tensile testing	Contact details of Testing lab
Currently available testing	Tensile Compressive Shore Hardness	
Existing data can be found on our Granta Mi materials database	Link to Granta Mi Database	

Figure 7: AM Material Characterization Guide for Designers aide memoir

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REFERENCES

[1] Stratasys Ltd., 2020. ULTEM 9085 Spec Sheet. USA: Stratasys [23/07/2020]. Available from: https://www.stratasys.com/-/media/files/material-spec-sheets/mss_fdm_ultem9085_1117a.pdf.

[2] ULTEM 9085, 2020. Stratasys FDM materials, ULTEM 9085 Resin [23/07/2020] Available from: http://www.stratasys.com/materials/fdm/ultem-9085.

[3] HALLGRIMSSON B., 2012. Prototyping and Model making for Product Design, London UK: Laurence King Publishing Ltd

[4] Senvol LLC., 2020. Material Results. USA: Senvol [23/07/2020]. Available from: http://senvol.com/database/

[5] Stratasys Technical Application Guide for FDM Jigs and Fixtures. USA: Stratasys, 2020 [23/03/2020]. Available from: https://www.stratasys.com/-/media/files/education-f123/tags/tag_fdm_jigsfixtures_en_1015_web.pdf?la=de&hash=74EFCE506C1EAE8B0B61B4716F730106FC5897FB

[6] CAFFREY, T., WOHLERS, T. and CAMPBELL, R.I., 2015. Wohlers Report, 3D Printing and Additive Manufacturing State of the Industry Annual Worldwide Progress Report 2015. Fort Collins, Colorado: Wohlers.

BIOGRAPHIES

"**Dr. Fayyaz Rehman** is an Associate Professor at Warsash School of Maritime Science and Engineering, Solent University, UK. He is a Fellow of Higher Education Academy, a Chartered Engineer from the Engineering Council and a Fellow of the Institution of Engineering Designers, UK. He is also vice chair and committee member of the Consortium of UK Manufacturing Engineering Heads (COMEH), a UK-based body responsible for promoting manufacturing engineering education and research, as well as organizing the International Conference on Manufacturing Research (ICMR) conference series annually. His research interests are CAD/CAM/CAE, Material Testing and Additive Manufacturing Technologies."

"**John Diston** is a Manufacturing Engineer with experience of supplying additively manufactured components and materials data in aerospace and motorsport sectors and continually influences users by promoting its adoption and best practices in both design and manufacture. He has contributed to the International Conference on Manufacturing Research (ICMR) conference series."