Characterization of Electrically Conductive 3D Printing Materials for use in ESD Control Programs

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Abstract –3D printing is quickly becoming a widely used additive manufacturing technique in the rapid prototyping of items for use within an electrostatic discharge (ESD) control program. Many organizations developing electrically conductive 3D printed materials may not have the necessary equipment or expertise to sufficiently evaluate them. This paper characterizes various materials and 3D printing parameters that may influence the final product's electrical performance.

I. Introduction

Additive manufacturing processes, such as three-dimensional (3D) printing, continue to gain popularity as the technology advances. The 3D printing market size was estimated at USD 13.84 billion in 2021 and is expected to grow to USD 62.76 billion by 2030 [1].

This paper focusses on the fused deposition modeling (FDM) process, also known as fused filament fabrication (FFF), which is a common type of 3D printing technology used at the consumer level due to its affordability and availability.

Numerous companies have begun developing advanced composites that enhance the base material properties [2] – [5]. Commonly, the base material is a type of thermoplastic (i.e., acrylonitrile butadiene styrene (ABS), polylactic acid (PLA), polyethylene terephthalate glycol (PETG), etc.) which is loaded with another material (e.g., carbon black, carbon fiber, glass fiber, etc.) to provide additional properties such as electrical or thermal conductivity or increased mechanical strength.

A. Introduction to FDM / FFF

An FDM printer consists of a few key components; material (usually called filament, contained on a spool), an extruder, and a build platform – as shown in Fig. 1. The filament is fed from the filament spool into the extruder until it reaches the heated extrusion head. The extruder is mounted on a three-axis system allowing for three-dimensional (x, y, and z) movement. As the filament is pushed into the heated extrusion head it begins to melt and is dispensed onto the build platform to apply the first layer. Multiple passes are required whereby the material is continuously added layer by layer resulting in a finished part.

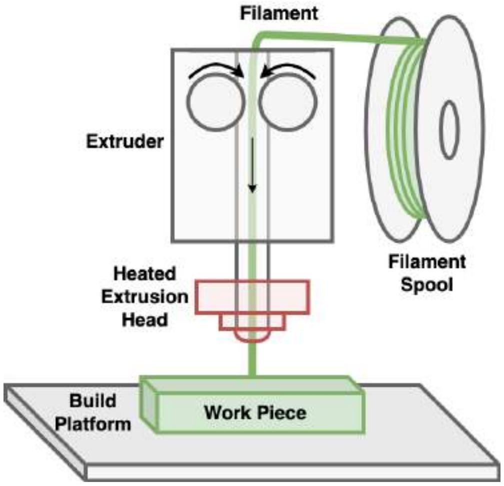

Figure 1: FDM Printer Schematic

There are numerous parameters involved in 3D printing, ranging from material selection, product design, and printer settings – each may drastically change the result of the final product.

B. Filament Industry Review

Multiple 3D printing companies now offer 'ESD safe' filaments [6] – [8]. Often the materials are evaluated using ASTM D257 [9] which may not be suitable for evaluating the resistive properties of materials intended for use within an ESD control program.

Some companies do test in accordance with ESD Association [10] Standard Test Methods; however, it is not common for the printer's parameters to be included with the data sheet or testing results.

Ultimately, this results in a disconnect between the datasheet and real-world measurement data due to either differences in test methods or printing parameters.

II. Specimen Preparation

A. Specimen Design

Test specimens were designed to evaluate the two major print directions, horizontal (H) (x & y axis) and vertical (V) (z axis). This was achieved by creating a 100mm × 100mm × 4mm surface, with 100 % infill, printed both horizontally and vertically with a small support structure – see Fig. 2.

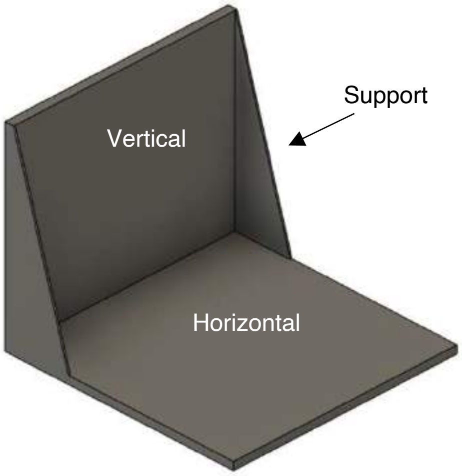

Figure 2: Specimen Design CAD Model

The specimen's horizontal surface has a diagonal pattern as seen in Fig. 3, and the vertical surface has a horizontal pattern as seen in Fig. 4.

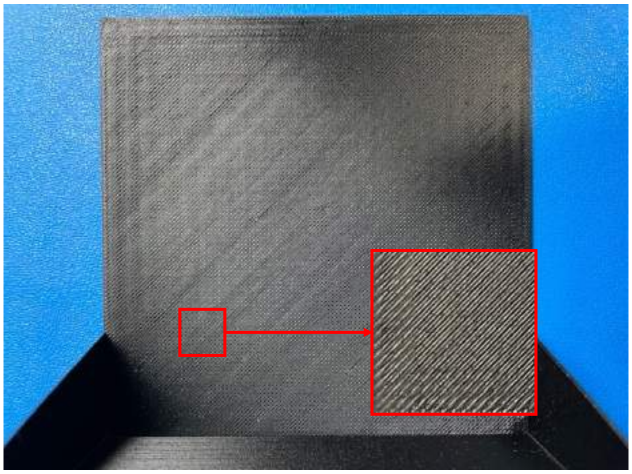

Figure 3: Horizontal Surface with Diagonal Pattern

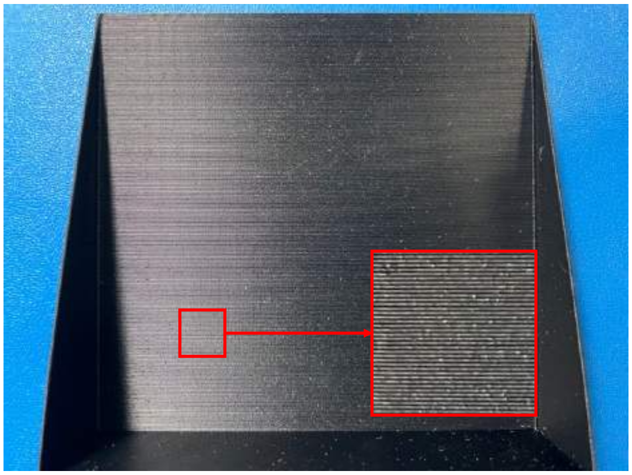

Figure 4: Vertical Surface with Horizontal Pattern

B. Printer Hardware

All specimens were created with a MakerGear M3 desktop 3D printer [11] as shown in Fig. 5.

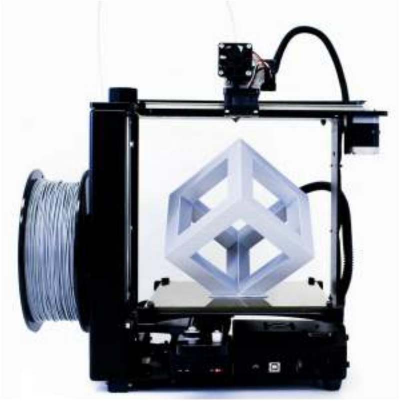

Figure 5: MakerGear M3 3D Printer [11]

C. Filament Properties

All materials evaluated in this paper are from 3DXTECH's 3DXSTAT filaments [8] which use multi-walled carbon nanotubes (MWCNT).

Carbon atoms are hexagonally bonded together in a single-layer sheet to create graphene. Graphene can then be rolled into a tube to create a carbon nanotube. Multiple layers of graphene can be rolled into concentric tubes to create the multi-walled carbon nanotubes – see Fig. 6.

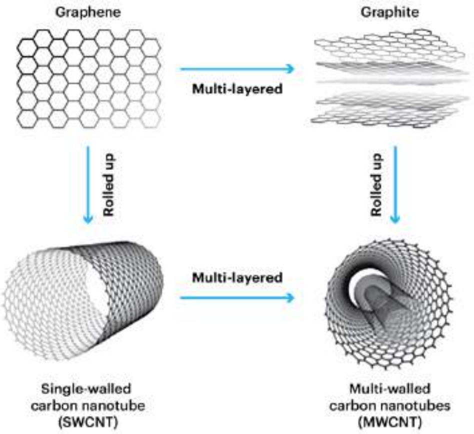

Figure 6: Graphene to MWCNT Process [12]

There is evidence to suggest that MWCNTs provide increased conductivity per loading percentage compared to other loading materials (e.g., carbon black, graphene, etc.) [13].

III. Test Methods

All testing was conducted at ambient conditions with temperature of (21 ± 2) °C and relative humidity of (44 ± 5) %.

A. Surface Resistance

Surface resistance is a critical parameter within an ESD control program, especially for materials that will directly contact ESD sensitive (ESDS) items. This allows for the surface to be grounded and/or if a charged ESDS item discharges into the material the current flow can be controlled.

1. ANSI/ESD STM11.11

Specimens were measured in accordance with ANSI/ESD STM11.11-2021 [14] to evaluate their surface resistance – see Fig. 7.

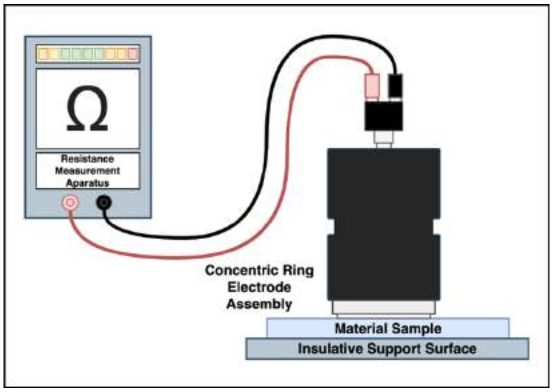

Figure 7: ANSI/ESD STM11.11 Set Up

An average of five measurements per horizontal and vertical surface is presented.

2. ANSI/ESD STM11.13

Specimens were measured in accordance with ANSI/ESD STM11.13-2021 [15] to evaluate localized surface resistance – see Fig. 8.

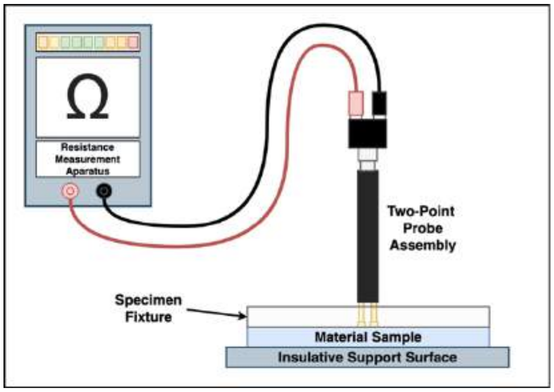

Figure 8: ANSI/ESD STM11.13 Set Up

In order to ensure consistency between specimens, a small test fixture was created from insulative material which fits between the specimen support walls and allows for the two-point probe assembly to fit through.

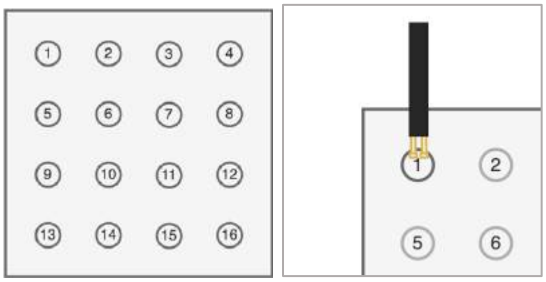

Figure 9: Specimen Fixture Locations

Data was gathered from corner locations (#1, 4, 13, 16) from Fig. 9, unless otherwise specified.

The two-point probe assembly was always oriented the same way resulting in measurements across or along the dispensed bead.

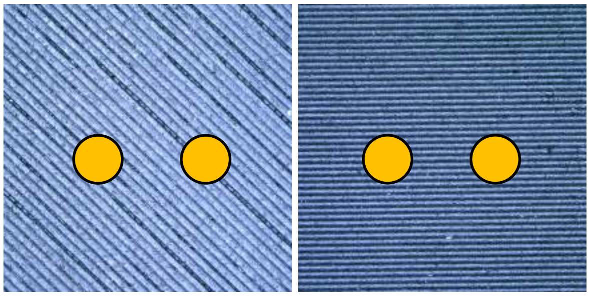

Figure 10: ANSI/ESD STM11.13 Measurement Location
Left is horizontal surface, Right is vertical surface

B. Volume Resistance

Like surface resistance, volume (bulk) resistance is another critical parameter within an ESD control program, especially for materials that are to be electrically bonded or grounded.

Specimens were measured in accordance with ANSI/ESD STM11.12-2021 [16] to evaluate their volume resistance – see Fig. 11.

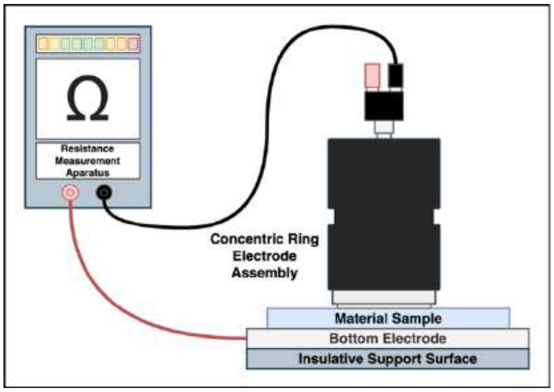

Figure 11: ANSI/ESD STM11.12 Set Up

An average of five measurements per horizontal and vertical surface is presented.

IV. Experiment 1 – Extrusion Temperature

A. Specimen Preparation

Four specimens of each PLA and PETG were printed with a 0.4 mm extrusion nozzle diameter across a range of extrusion temperatures resulting in a total of eight.

Table 1: Specimen Numbers

	Extruder Temperature [°C]						
Material	210	220	230	240	250	260	270
PLA	1	2	3	4			
PETG				5	6	7	8

B. Surface Resistance

1. ANSI/ESD STM11.11

The PETG specimens show a clear relationship between extrusion temperature and surface resistance – as temperature increases the resistance decreases – from 3E10 Ω at 220 °C to 3E7 Ω at 260 °C.

The PLA specimens did not follow this trend with the 220 °C specimen being an outlier measuring between 2E7–4E7 Ω. The remaining specimens (230–250 °C) did trend downward with increased temperature; however, they show a significant difference between the horizontal and vertical surface resistance measurements.

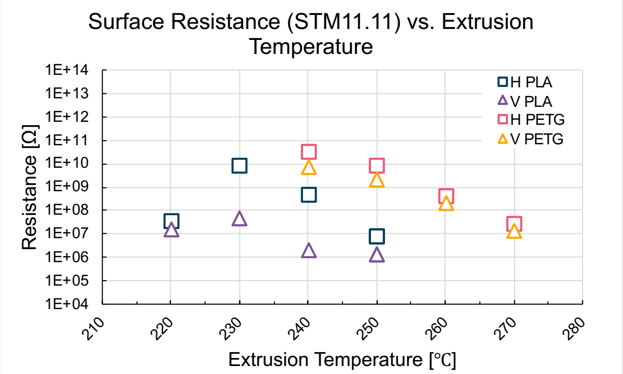

Figure 12: Surface Resistance vs. Extrusion Temperature

A second set of specimens were printed for Experiment 2. Five PLA specimens were printed with the same printer parameters as Experiment 1 and were measured to verify the original data.

Fig. 13 shows that these PLA specimens do have a strong relationship between surface resistance and extrusion temperature measuring 3E13 Ω at 220 °C and 1E8 Ω at 260 °C.

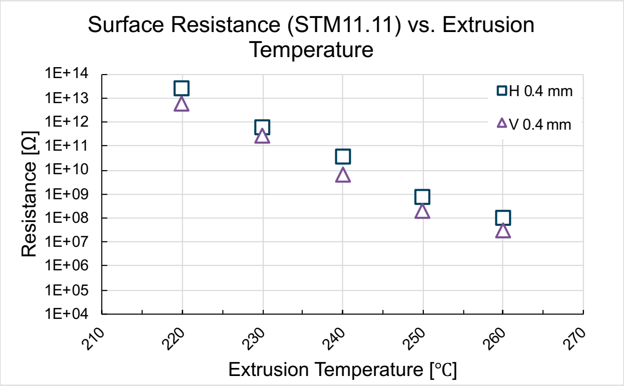

Figure 13: Second Specimen Set Surface Resistance vs. Temperature

This data indicates there may be part-to-part variability resulting from the raw material and/or printing quality.

2. 3DXTECH Evaluation

3DXTECH had previously conducted a similar evaluation of surface resistance vs. extrusion temperature across a range of their materials [17]. The results of their work have been summarized in Fig. 14.

The 3DXTECH data shows there is a strong relationship between extrusion temperature and surface resistance regardless of what kind of MWCNT loaded material is used.

3. ANSI/ESD STM11.13

Data from STM11.13 evaluation follows STM11.11 data trends as shown in Fig. 15. PETG consistently trends to a lower resistance with increased temperature – from 1E11 Ω at 240 ℃ to 3E7 Ω at 270 ℃.

Whereas the PLA trend is thrown off again by the 220 ℃-specimen – results are between 4E8 Ω at 230 ℃ and 2E6 Ω at 250 ℃.

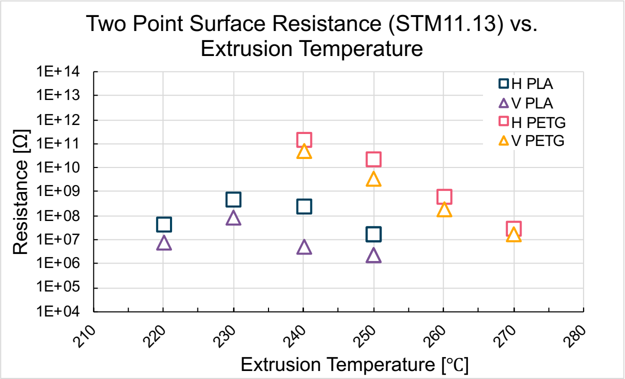

Figure 15: Two-Point Surface Resistance vs. Extrusion Temperature

The measurements were repeated with the additional PLA specimens from Experiment 2 and a clearer trend can be seen in Fig. 16. Resistance is almost linear from 2E12 Ω to 1E8 Ω across the temperature range.

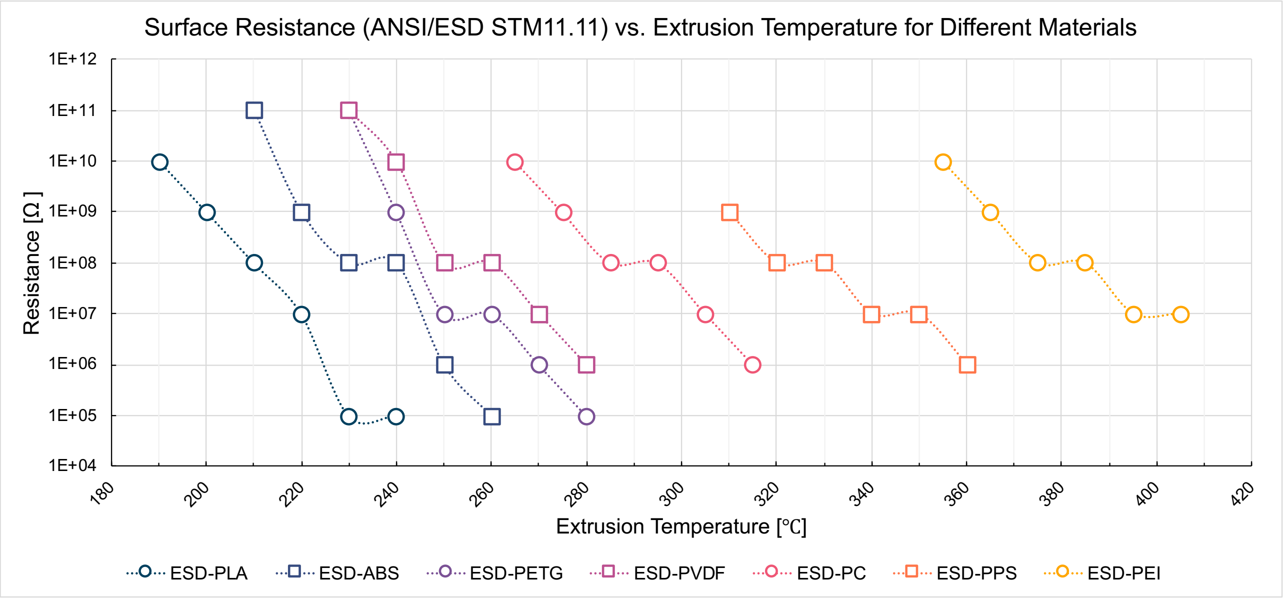

Figure 14: Summarized 3DXTECH Data [17]

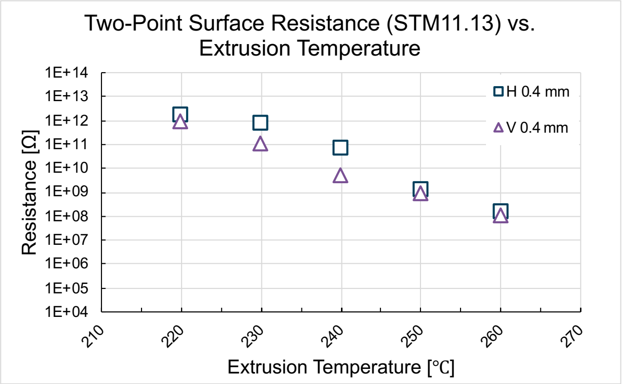

Figure 16: PLA Two-Point Surface Resistance vs. Extrusion Temperature

C. Volume Resistance

Like the surface resistance data, the PETG specimens have a strong relationship between extrusion temperature and volume resistance per ANSI/ESD STM11.12 – see Fig. 17.

The vertical surface's volume resistance is approximately one order of magnitude lower than the horizontal surface. This is likely due to the relatively high surface resistance of the first layer of the print which requires preparation to the surface.

The PLA results from the vertical surface show a strong relationship, however the horizontal surface's volume resistance varies greatly. This is also likely due to the initial print layer.

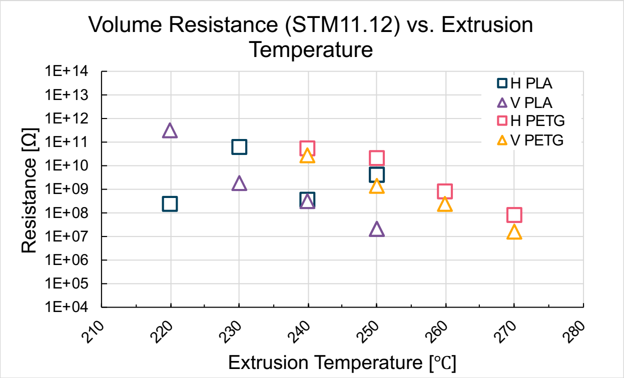

Figure 17: Volume Resistance vs. Extrusion Temperature

Measurements were repeated with specimens from Experiment 2 and a clear trend is seen in Fig. 18.

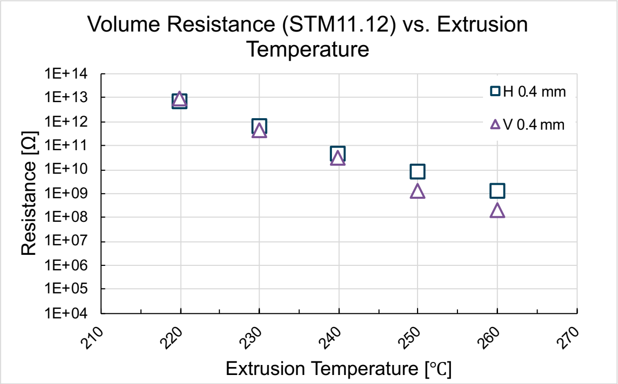

Figure 18: PLA Volume Resistance vs. Extrusion Temperature

V. Experiment 2 – Nozzle Diameter & Layer Height

A. Specimen Preparation

Five specimens of PLA were printed with a 0.4 mm and a 0.6 mm extrusion nozzle diameter, across a range of extrusion temperatures resulting in a total of ten specimens.

Table 2: Specimen Numbers

Nozzle Diameter [mm]	**Extruder Temperature [°C]**				
	220	230	240	250	260
0.4	1	2	3	4	5
0.6	6	7	8	9	10

B. Surface Resistance

1. ANSI/ESD STM11.11

0.6 mm diameter extrusion nozzle specimens have significantly reduced surface resistance. The 0.6 mm specimens measure 7E7 Ω at 220 °C and 5E5 Ω at 260 °C. The 0.4 mm specimens measure 3E13 Ω at 220 °C and 1E8 Ω at 260 °C.

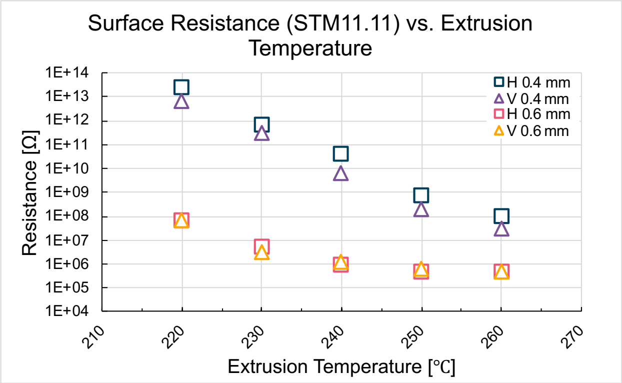

Figure 19: Surface Resistance vs. Extrusion Temperature

1. ANSI/ESD STM11.13

The STM11.13 measurements show the 0.6 mm specimens being orders of magnitude lower resistance than the 0.4 mm specimens – 1E8 Ω at 220 °C and 3E5 Ω at 260 °C. Comparatively, the 0.4 mm specimens measure 2E12 Ω at 220 °C and 1E8 Ω at 260 °C.

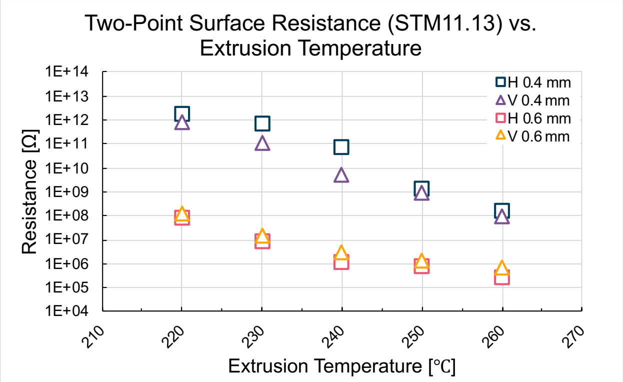

Figure 20: Two-Point Surface Resistance vs. Extrusion Temperature

2. Surface Consistency

Four specimens (#1, 5, 6, 10 from Table 2) were further evaluated per ANSI/ESD STM11.13 by measuring all 16 locations of the fixture (see Fig. 9) on the specimen's horizontal surface.

The 0.4 mm 220 °C specimen measures between 3E11 – 2E12 Ω, showing the largest variation of the specimens.

The 0.4 mm 260 °C and 0.6 mm 220 °C specimens measure similarly, showing an approximate half order of magnitude difference between minimum and maximum values.

The 0.6 mm 260 °C specimen is the most consistent with minimal variation.

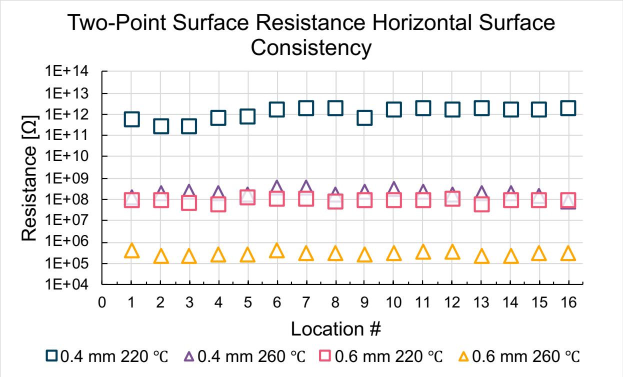

Figure 21: Two-Point Surface Resistance Consistency

3. Two-Point Probe Assembly Orientation

The same four specimens were again evaluated to determine the influence of the two-point probe assembly's orientation against the print direction. All previous data had the probe oriented per Fig. 10, the following data compares the probe oriented parallel and perpendicular to the print direction. Data was gathered from corner locations (#1, 4, 13, 16) from Fig. 9.

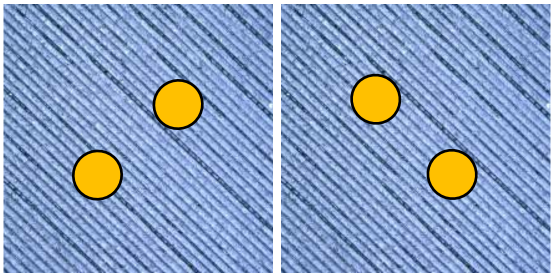

Figure 22: Two-Point Probe Orientation
Left is perpendicular, Right is parallel

The data indicates probe orientation does not significantly influence the result with less than one order of magnitude difference between orientations as shown in Fig. 23.

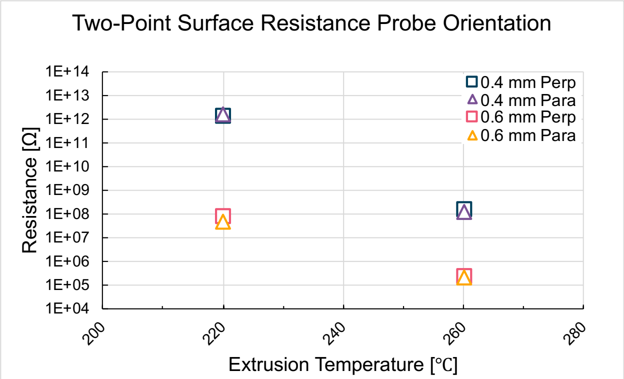

Figure 23: Two-Point Probe Orientation Surface Resistance

C. Volume Resistance

Specimens were measured in accordance with ANSI/ESD STM11.12, the data shown in Fig. 24 shows similar trends to surface resistance data as seen in Fig. 19 and 20.

The 0.4 mm specimens measure 1E13 Ω at 220 °C and 2E8 Ω at 260 °C and have less than one order of magnitude difference between the horizontal and vertical surfaces.

The 0.6 mm specimens measure 2E10 Ω at 220 °C and 1E6 Ω at 260 °C. The data also shows a significant difference, approximately two orders of magnitude, between the horizontal and vertical surfaces between 220–250 °C.

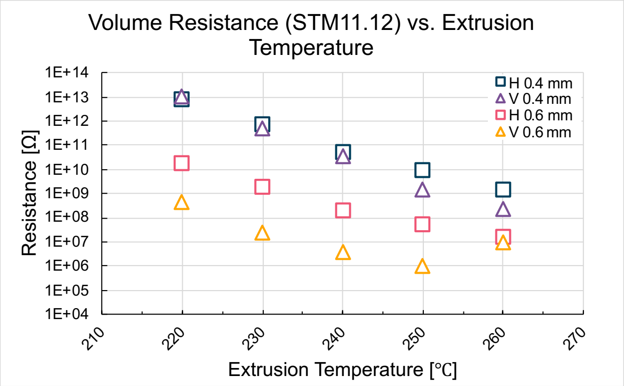

Figure 24: Volume Resistance vs. Extrusion Temperature

The variation between surfaces is, again, likely due to the preparation of the first layer.

D. Resistance to Ground

To evaluate the grounding / bonding capabilities of the material a resistance to ground (R_g) test set up based on ESD TR53 [18] was developed.

The specimen was mounted onto an insulative support surface and bonded to the equipment grounding conductor (ground) via a stainless-steel strip measuring 12 × 76 mm clamped on the specimen's vertical surface to improve bonding.

A resistance measurement apparatus was connected to the equipment grounding conductor, and to a resistance measurement electrode. The electrode was placed centered on the specimen's horizontal surface.

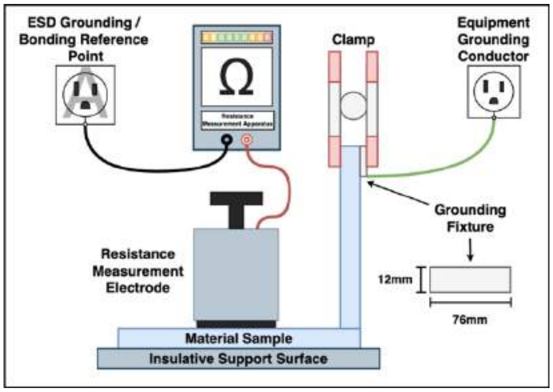

Figure 25: Resistance to Ground Set Up

The 0.4 mm specimens range from 7E11 Ω at 220 °C to 3E8 Ω at 260 °C.

The 0.6 mm specimens range from 8E8 Ω at 220 °C to 7E5 Ω at 260 °C which would make them suitable for control of ESD.

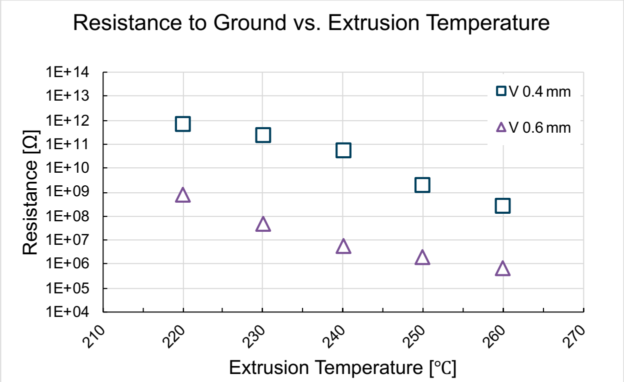

Figure 26: Resistance to Ground vs. Extrusion Temperature

VI. Conclusion

Some of the many 3D printing materials and printer parameters were studied. It is clear there is opportunity for the use of electrically conductive 3D printed materials within ESD control programs. However, it is important to understand the variability in the final product.

The correct material must be selected for the application (e.g., tighter dimensional tolerance, ability to withstand hot or cold temperatures), most importantly be suitable for use in proximity or direct contact to ESDS items.

The data presented shows that there is a relationship between the printer's extrusion temperature and the specimen's surface and volume resistance – as temperature increases the resistance decreases.

The end user must be aware of the material's extrusion temperature range, if the temperature is too high there may be additional negative effects on the final product, such as poor print quality.

It was found that there can be part-to-part resistance variability that may be a result of several factors, including but not limited to the material batch, storage, and handling, and the printer's parameters or printing environment.

Increasing the nozzle diameter from 0.4mm to 0.6mm significantly influences the specimen's resistance, reducing the resistance by at least one order of magnitude, if not more, across the temperature range. This is likely due to the resulting increase in layer height, increasing both surface area of the print bead and reducing the number of layers. It is suspected that each layer provides an opportunity for a poor electrical connection, increasing resistance.

Resistance also varies between the print direction; end users should be aware of this factor and take it into account when designing and printing products for use within their program.

VII. Further Discussion

A. 3D Printing Opportunities in an ESD Control Program

Conductive and dissipative materials are critical in the mitigation of ESD risks, and many are used in the construction of resistive control items within an ESD control program, such as packaging, worksurfaces, and tools / equipment.

While electrically conductive 3D printed items may not be a direct replacement for all currently available ESD control items they are well suited for rapid prototyping, and small quantity manufacturing. Some examples include packaging, enclosures, hand tools, and fixtures as seen in Fig. 27 and 28.

Figure 27: Various 3D print applications by Formlabs [19]

Figure 28: Electronics enclosure by 3DXTECH [8]

B. Surface Finish

Some of the specimens were inspected under 100 x magnification – see Fig. 29 and 30. The bead thickness from the 0.6 mm nozzle diameter is larger compared to the 0.4 mm specimens.

Additionally, the 0.6 mm specimens appear to have more consistent fusing between the beads compared to the 0.4 mm specimens which have gaps. These gaps may be a contributing factor to the increased resistance of the 0.4 mm specimens.

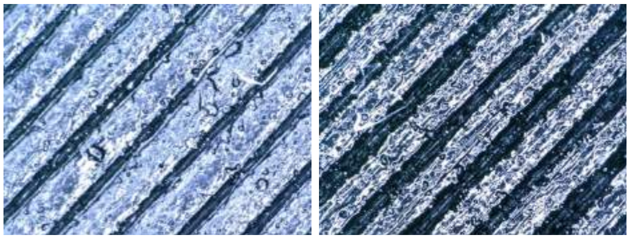

Figure 29: PLA 0.6 mm 100x Zoom Horizontal
Left is 220 °C, Right is 260 °C

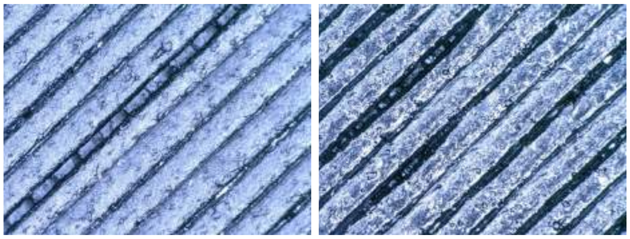

Figure 30: PLA 0.4 mm 100x Zoom Horizontal
Left is 220 °C, Right is 260 °C

C. Future Studies

This paper presents a limited evaluation of electrically conductive 3D printed materials. Further work can be done to better understand the wide range of materials and printing parameters that influence the final product. Examples include but are not limited to:

1. Voltage Dependency

Previous work [20, 21] has shown that test voltage influences the measured resistance. This paper follows industry standard test methods, however further evaluation of voltage dependency may provide beneficial real-world insight.

2. Humidity Dependency

Previous work [20] has shown that relative humidity influences the measured resistance.

This paper's experiments were done at ambient conditions, evaluation at lower relative humidity (e.g., 12 % ± 3 %) would be beneficial to understand the specimen's performance in a worst-case environment.

3. Printer Infill Percentage

The density of a 3D print relates to the infill percentage parameter. With increased density there are more electrical connections within the bulk of the material. All testing in this paper was done with 100 % infill. Evaluation of materials across a range of infill (10 %, 25 %, 50 %, 75 %, etc.) would provide insight into more optimized designs (e.g., lighter weight, less material usage, etc.)

VIII. Acknowledgements

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