Exploring the Relationship Between Dual Material Filament Annealing Temperature and Material Properties

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SUMMARY

The use of additive manufacturing (e.g., AM) in research, industrial, and hobbyist environments has expanded rapidly in recent years. This has resulted in a massive increase in filament types and their potential combinations available for AM. For many of these new materials, the best processing and post-processing conditions are not fully understood. Annealing at high temperatures has been proposed as a method to modify the mechanical properties of dual-material filaments, like tensile strength, but early results suggest that changes may be minor. This research sought to evaluate annealing temperatures' relationship to tensile strength. For high annealing temperatures, we hypothesized that annealing is negatively correlated with tensile strength. To test this theory, dogbones were printed using a filament with a polycarbonate core and acrylonitrile butadiene styrene shell and annealed at different temperatures. The annealed dogbones were tested in a tensile test machine to evaluate the annealed materials' tensile strength. This study found that annealing at 95°C did not significantly increase the tensile strength, while annealing at 135°C decreased tensile strength. However, annealing at 95°C significantly reduced the overall variation in tensile strength (n=10). Further research is needed to determine the precise relationship between annealing temperature and tensile strength of dual material filaments, as well as to evaluate other potential relationships to material properties such as bending strength.

INTRODUCTION

Additive manufacturing (AM), more commonly known as 3D printing, has emerged as a revolutionary production technology in industrial manufacturing and prototyping. Unlike subtractive manufacturing methods, AM produces objects by laying down layers of material that exactly match a digital model. AM is faster, less expensive, more flexible, and less wasteful than traditional subtractive methods [1]. AM can also employ a variety of materials including plastic, gel, and metal [2]. Industrial and research-grade AM filaments have expanded to include self-healing polymer designs to overcome vertical (z-level) degradation, hydrogels [3], and multimaterial filaments composed of woods, polymers, ceramics, and metals [4]. However, most current hobbyist-grade AM machines rely on plastic filaments. This limits a product's achievable structural strength to what plastic filaments can achieve. Common plastic filaments include acrylic, polylactic acid (PLA), acrylonitrile butadiene styrene (ABS), thermoplastic polyurethane (TPU), and others. While each of these materials offers unique advantages, they also have disadvantages. For example, PLA has high tensile strength but weak temperature resistance [5]. In addition, these common filaments produce prints with low z-level layer adhesion [6].

One advancement used to improve layer adhesion is annealing. In annealing, components are heated to a temperature below their melting point but high enough to allow polymer chains to relax. This process relieves internal stresses that can make failures more likely, also improving other mechanical properties such as tensile strength. Plastics are typically annealed close to their glass transition temperature (T_g). A material's T_g temperature is the point at which a polymer's chains relax and the material transitions from a glassy state to a rubbery state [7]. Annealing near a material's T_g allows the polymer chains to undergo maximum relaxation without deforming, reducing stresses built up during the printing process and improving *z*-level adherence [8]. Plastics are not typically annealed over their T_g as excessive rearrangement of the polymer chains can cause structural deformations such as creeping and sagging, reversing annealing's effects. However, research has shown that annealing plastics above their T_g increases fracture toughness. For example, when annealed at 135°C, ABS has demonstrated a 27-fold increase in fracture toughness [9].

Dual material filaments have been proposed as a method for allowing annealing at higher temperatures. In these compositions, the more thermally resistant material provides structural integrity, allowing annealing to be conducted very close to the higher T_g of the composite. ABS has a T_g of roughly 100°C [10] and polycarbonate (PC) has a T_g of roughly 145°C [11], but testing conducted on ABS-PC composites annealed at 130°C revealed little improvement in tensile strength [12]. However, this could be attributed to the different thermal properties of the composite materials. ABS has a coefficient of thermal expansion (CTE) of 82.8 μm/m-°C [13], compared to 64.9 μm/m-°C for PC [14]. As such, ABS expands 28% more than PC. When annealing, this difference in CTE could introduce internal stresses that counteract the relief of stresses from annealing.

This work sought to clarify the relationship between annealing temperature and tensile strength of an ABS-PC composite. We hypothesized that annealing temperature has a negative relationship with tensile strength. This work found no major change in tensile strength for the specimens annealed at lower temperatures and a decrease for the specimens annealed at higher temperatures. We also found that annealing at lower temperatures decreased the variation in tensile strength. While the results were inconclusive, they may indicate that one of the reasons that annealing DM filaments did not increase the tensile strength as expected is they were annealed at too high a temperature.

RESULTS

This study's objective was to compare the change in tensile strength from annealing at temperatures above and below the lower T_g of the composite. To do this, we produced three different sets of identical specimens composed of a acrylonitrile butadiene styrene-polycarbonate (ABS-PC) composite. The control group was left unannealed, while the other groups were annealed at 95°C and 135°C respectively. The specimens were tested to determine their peak load strength. The data was normalized by cross-sectional area to determine maximum tensile strength. The data from these tests indicates the maximum force that the material can withstand before failure (**Table 1**). If annealing had a negative relationship with tensile strength as we hypothesized, its maximum tensile strength would be lower than the control group.

Test Number	Test Group		
	Control Tensile Strength (mPa)	95°C Annealing Tensile Strength (mPa)	135°C Annealing Tensile Strength (mPa)
1	33.64	34.47	30.98
2	36.25	36.95	30.24
3	35.96	35.96	32.73
4	31.99	35.6	31.24
5	34.81	34.22	29.11
6	37.01	34.32	31.73
7	31.91	34.87	32.05
8	33.47	36.14	31.88
9	36.38	35.65	32.43
10	34.6	35.35	31.38
Av.	34.60	35.35	31.38
Sdev	1.66	0.88	1.08

Table 1. Tensile Strength (mPa) of Test Specimens. Ten dogbones from each testing group were tested using the tensile strength testing machine described in the "Materials and Methods" section. Each of the specimens' maximum tensile strength is shown in megapascals. The average value and standard deviation for each test group are also included.

There was a statistically significant difference among the three groups' tensile strength (one-way ANOVA, Welch correction, $p<0.001$, $\eta^2_p = 0.654$, **Figure 1**). The large effect size indicates that approximately 65% of the variance in tensile strength between groups is attributable to the annealing temperature. There was a significant decrease between the tensile strength of the 135°C group and both the control and the 95°C group (Games-Howell, $p<0.001$), but no significant difference between the control group and the 95°C group (Games-Howell, $p=0.489$). These findings suggest that annealing at 95°C did not significantly increase tensile strength, while annealing at 135°C decreased the tensile strength.

However, annealing did significantly reduce the variance in the 95°C group (Levene's Test, $p=0.032$), but not amongst the 135°C group ($p=0.075$).

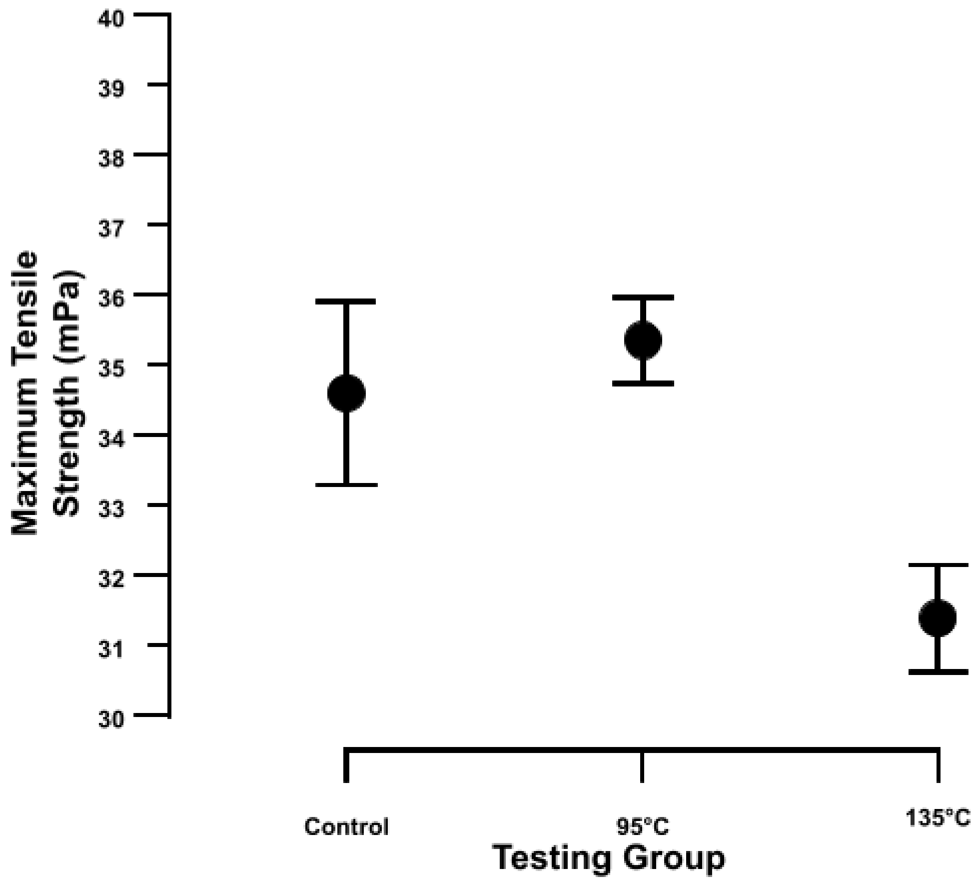

Figure 1. Average Maximum Load Strength of Test Groups in mPa. A scatter plot with error bars based on the average maximum load strength of each test group and standard deviation in megapascals. The data was collected using the tensile testing machine described in “Materials and Methods”. The graph was created using JASP.

DISCUSSION

This study evaluated whether a high annealing temperature has a negative relationship for the tensile strength of filament composed of a PC core and ABS. The results indicate that annealing at 95°C did not significantly increase maximum tensile strength, while annealing at 135°C decreased tensile strength. This suggests that annealing temperature beyond a certain threshold has a negative correlation with tensile strength.

Since the ultimate stress was calculated using the CAD model dimensions, there are multiple sources of dimensional error that should be noted. The Voron 2.4 was measured to be accurate to within 0.1 mm on the *x*- and *y*-axes and 0.2 mm on the *z*-axis using calibration cubes and a digital caliper. The CAD dimensions of the gauge area were 2.8 mm wide and 3.3 mm high with a total area of 9.18 mm^2 due to chamfered corners. Given the dimensional variation of the printer, the estimated minimum and maximum area was 8.06 mm^2 and 10.5 mm^2, respectively. This resulted in an area variation between -1.12 mm^2 and 1.32 mm^2. When propagated to the stress measurements, this resulted in an uncertainty of 4.36 mPa for the control, 4.46 mPa for the 95°C group, and 3.96 mPa for the 135°C group.

Another source of dimensional error is the annealing process. The cross-section measurements did not account for potential deformation caused by the annealing process, which was especially evident in the 135°C group (**Figure 2**). The sagging of specimens in the 135°C group may have decreased the gauge area, decreasing the peak load strength of the specimens. Since our calculations assumed that all gauge areas would be equal, this inaccuracy may have resulted in an underestimate of the tensile strength of the 135°C group. Future studies should explore methods of accurately assessing gauge area after annealing, such as photogrammetry, or consider excluding specimens with significant sagging.

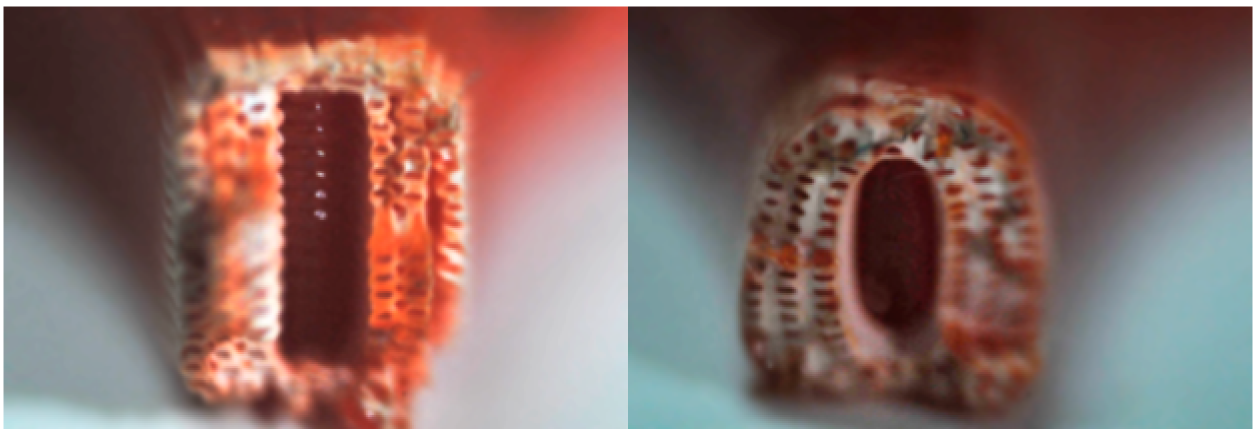

Figure 2. Cross Sections of Annealed Specimens. On the left is the close-up cross-section of a specimen annealed at 95°C after failure. On the right is a close-up cross-section of a specimen from the 135°C group after failure. The 135°C specimen suffered more creep and sagging than the 95°C specimen.

The sagging exhibited in the 135°C group indicates that although annealing has been proposed as a method for increasing tensile strength, a limit exists that mitigates these effects due to the deformation of the ABS-PC material when annealed at higher temperatures. However, the 95°C specimen retained its structural integrity. It would be worthwhile to compare the structural retention of a purely ABS specimen with the 95°C specimen. If the ABS specimen has more deformation, it would validate the hypothesis that DM filament enables annealing at higher temperatures without compromising tensile strength. The structural deformations of the 135°C specimen could in part be due to unequal thermal expansion rates of the composite materials. Additional tests should be performed with a composition with a higher T_g but similar or greater difference in CTE to evaluate the extent that the different expansion rates impact the specimens when thermal deformation is ruled out. Additional tests could also be performed with ABS-PC specimens annealed between 95°C and 135°C to determine if there is an optimal temperature that increases tensile strength without deforming specimens.

The annealing process may have also impacted the results. The specimens were annealed in a toaster oven. The toaster oven did not have precise temperature controls, and the environment fluctuated in temperature by an estimated maximum of 5° C. Therefore, the dogbones were not annealed consistently at the desired temperature. The toaster oven was chosen because it is realistic and affordable. It was important not to use a food-grade oven because annealing plastics can cause outgassing of carcinogens, such as formaldehyde and styrene, that contaminate the oven [15].

Error could also have originated from the luggage scale used to measure the force applied to samples. The luggage scale was advertised as accurate to within 25 grams and having a frequency of 5Hz. These factors could have resulted in slight inaccuracies in the maximum load strength reading.

As a result of these various sources of error, this study cannot definitively support the hypothesis that annealing temperature of a DM specimen is negatively correlated with tensile strength. Further studies using a different annealing process should be conducted to verify our findings. Also, experiments should be done with different DM compositions. PLA-PC represents a promising option because the delta T_g between the composite materials is around 90°C, significantly more than the 55°C difference between ABS-PC. Testing this material combination would help ascertain if the DM structure does help retain structural integrity beyond the lower material's T_g and the role of differential thermal expansion in deformations.

However, this study made the discovery that annealing at 95°C may decrease the variation in tensile strength. This is a valuable finding for the world of AM, especially as its uses have expanded beyond prototyping and towards large-scale production. Consistency is important for large-scale manufacturing, and annealing is a way for manufacturers to increase consistency without sacrificing tensile strength. This may be important in applications with low tolerances, such as 3D printed jigs and fixtures in the aerospace industry. Further studies should be conducted to verify that annealing decreases variation in DM specimens and whether its benefits extend to single filament specimens. These studies should also have larger sample sizes to confirm the significance of the decrease in variance.

This study has also proven the viability of a tensile testing machine that is affordable and accessible to hobbyists. This is in line with the open-source culture of the hobbyist creator community. As more types of filaments are released, hobbyist makers will require a method of delineating between filament types to determine which material best suits their application. Our tensile testing machine represents an easy, affordable method for makers to test materials' tensile properties for themselves. While it has not been proven that annealing DM filaments increases tensile strength, it has shown that annealing may decrease tensile strength variance as well as enable the hobbyist community to conduct further research.

MATERIALS AND METHODS

A mid-level 3D printer (Voron 2.4) was used to create a dual-material spool. Using the dual head of the printer, a spool was printed using a PC (Bambu Labs PC) core and an outer shell of ABS (Polymaker PolyLite™ ABS) (**Figure 3**). PC was chosen as the core material because it is generally stiffer than ABS (5). As a result of the difference in ductility, the PC often fractured, whereas the ABS underwent plastic deformation (**Figure 4**). Once a spool of DM filament was printed, it was used as the loaded spool in the Voron to produce sets of dogbones. All dogbones had identical printing settings. Dogbones were 50 mm-long and had a gauge section area of 9.175 mm^2.

Once printed, the dogbones were divided into three test groups. The first group was unannealed and used as a test group. The second was annealed at 95°C for one hour in a toaster oven. The third group was annealed at 135°C for one hour. These temperatures were chosen due to the glass transition temperature (T_g) of the composite materials in the DM filament. ABS has a T_g of approximately 100°C (10) and PC has a T_g of approximately 145° C (11).

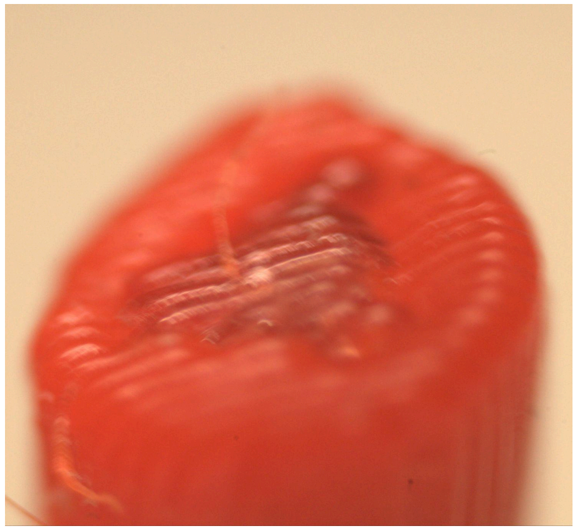

Figure 3. Close-up of DM Filament. A close up photograph of the second stage of the production process. The filament was printed on a 3D printer. After, it is fed into the same printer to produce dogbones.

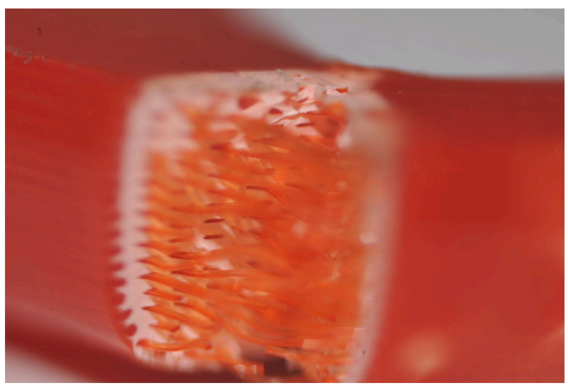

Figure 4. Unannealed Dogbone. A partially broken, unannealed dogbone. The red material is ABS plastic, and the white material is PC. The ABS underwent plastic deformation while the PC underwent brittle fracture.

The dogbones in the annealed test groups were heated to the desired temperature in a toaster oven. Both groups were heated gradually, left at the desired temperature for one hour, and then gradually cooled to room temperature. The gradual temperature change helped to prevent thermal shock.

Once the dogbones had been annealed, each group was tested for tensile strength. The testing machine consisted of a linear actuator (Pegelong, Cat# B0D3HD4G2N) and a digital luggage scale (American Weigh Scales, Cat# B0012TDR9E) (**Figure 5**). A dogbone was placed on a peg between the linear actuator and the scale, and the scale recorded the maximum force before the dogbone failed.

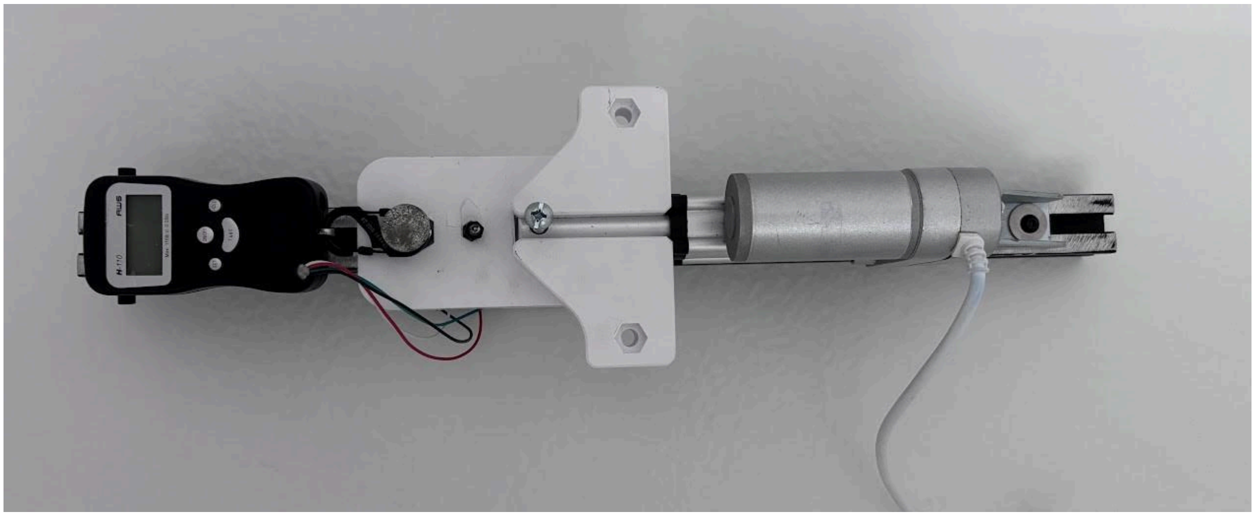

Figure 5. Tensile Testing Machine. A tensile testing machine composed of a linear actuator and a digital luggage scale. All of the parts are off-the-shelf or 3D printed.

To get reliable data, the load cell of the scale was extracted and wired to an operational amplifier (Elecrow HX711). The amplified voltage difference is wired into an Arduino (**Figure 6**).The Arduino was calibrated using three different objects with known weights to adjust the scaling factor. The Arduino processed the data and sent it through a serial port to a computer for data logging. This significantly increased precision with a standard deviation of less than 2 mPa in all test groups, compared to the 6.6 mpa standard deviation using the scale monitor readings.

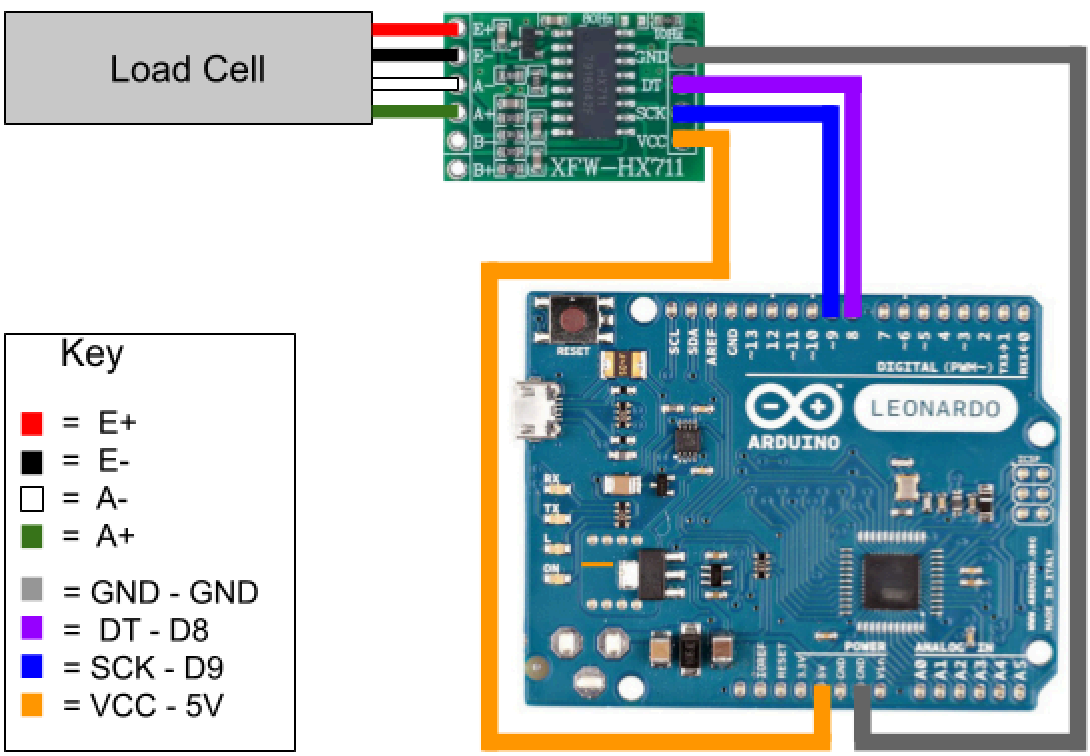

Figure 6. Arduino Wiring. The wiring diagram of the load cell of the digital luggage scale, the operational amplifier, and the Arduino.

The Arduino recorded data from the load cell 5 times a second. The data was graphed, and the maximum recorded load was converted from grams to newtons, then to Pascals using the known gauge section area of 9.175 mm^2 (9.175E-6 m^2). For each test group, ten specimens were evaluated until failure. The maximum tensile strength was recorded in mPa.

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