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The development of PVC extrusions for a 14,000 Ton self-supporting structure for the detection of neutrinos

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Abstract

The NOvA Neutrino Experiment has built a one of a kind self-supporting plastic structure, potentially the largest ever built. The PVC structure serves as a neutrino detector and is composed of 28 individual blocks that measure 15.5 m (51 feet) high by 15.5 m (51 feet) wide by 2.1m (7 feet) deep. The primary parts in the detector construction are 15.5 m (51-foot), 16-cell PVC extrusions. These extrusions form the basis of the detector modules which are laminated together in a crossed pattern to form the individual blocks and then filled with mineral oil based liquid scintillator. The self-supporting nature of the detector places important structural requirements on both the PVC formulation and the extrusions. Block assembly requirements impose narrow geometric tolerances. Due to the method of detecting neutrinos, the extrusions must possess exceptionally high reflectivity over a particular wavelength range. This requirement places additional restrictions on the components of the PVC formulation. Altogether, the PVC extrusions have to maintain important reflectivity characteristics, provide structural support to the detector, and meet relatively tight geometric requirements for assembly. In order to meet these constraints, a custom PVC formulation had to be created and extruded. We describe the purpose and requirements of the NOvA detector leading to the production of our unique PVC extrusion, summarize the R&D process, and discuss the lessons learned.

1 Introduction

A large and complex 16-cell PVC extrusion profile (Figure 1) has been developed for use as a neutrino detector for a physics experiment called NOvA [1]. The extrusion serves as the basic structural element of a "one-of-a-kind" self-supporting 15.5 m (51-foot) high structure, called a "Block", whose cells are filled with liquid scintillator containing approximately 95% mineral oil and 5% pseudocumene. A block contains 768 PVC extrusions in 32 layers and about 400,000 pounds of extruded PVC and is filled with 320,000 kg (700,000 pounds) of liquid scintillator. NOvA is comprised of 28 such Blocks with a total mass of 13,000 tonnes (14,000 tons) and size of 15.5 m (51 feet) high by 15.5 m (51 feet) wide and 59 m (194 feet) long.

It would have been preferred to make wider extrusions with 32 cells (1270 mm wide and 66 mm high) to match the data acquisition system. However, because of the challenge of making such a large profile with tight dimensional tolerances, it was decided to produce extrusions with 16-cells (635 mm wide by 66 mm high) and to subsequently join two extrusions side-by-side to form one "Detector Module", 15.5 m (51 feet) long and 1270 mm (50 inches) wide. To contain the liquid, modules are sealed on one end with a flat PVC plate and on the other end with a PVC manifold, which allows insertion and routing of fiber optics to transport the scintillation light to the photo-detectors.

The modules are assembled into a "Block Structure" by placing twelve modules side-by-side on a large mechanical platform, then placing a second layer of twelve modules on top of it, oriented perpendicular to the first layer. An adhesive is applied to the outer surfaces between the two layers to form a laminate. This overall process is shown in Figure 2. The fully-assembled Block is rotated via a mechanical platform and moved into position at the experimental hall.

2 The NOvA Detector

The NOvA experiment is located in Ash River, Minnesota, approximately 810 km away from the Fermi National Accelerator Laboratory (near Chicago), where a beam of neutrinos is created and aimed toward the Ash River site. The location was chosen specifically to study certain properties of neutrinos as they propagate 810 km underground. By analogy, as in a flashlight beam, the neutrino "spot size" gets broader with distance so that only a small fraction of the original neutrino beam produced at Fermilab is intercepted by the Ash River detector. Neutrino experiments require large detectors, and this experiment requires one of the biggest detectors in the world (and likely the largest self-supporting plastic structure in the world), yet at an affordable cost.

Neutrinos seldom interact with matter, but when they do ionizing radiation is a byproduct. The liquid scintillator generates light in the presence of ionizing radiation. Use of scintillating materials is common in nuclear and particle physics as well as other fields of research, including medicine, chemistry and biology. Owing to the very large volume of the NOvA detector, light produced deep within the detector cannot be detected directly. NOvA uses fiber optics running the length of each cell within the 15.5 m (51-foot) long module to capture the light and to transport it to avalanche-photodiode (APD) photo-detectors attached to the manifold covers (Figure 2). Since the fibers are very thin (0.7 mm in diameter), light may not strike a fiber immediately due to the small cross-sectional area of a fiber. The light is trapped within a PVC cell and bounces off the cell walls a number of times (averaging 8 bounces) before being captured by the fiber (Figure 3, Figure 4). The amount of light captured by the fiber optics is on average proportional to the eighth power of the reflectance. This "bouncing" is predominantly based on a diffuse reflection component, therefore a "shiny" or "glossy" PVC with a large specular-based component is not of help here. It is critical that the PVC surface does not absorb light, especially in the blue to near UV wavelengths, which is the color of the scintillation light. This drives the requirement of a highly reflective (non-absorptive) PVC extrusion.

3 Requirements for NOvA Extrusions

This extrusion profile must meet several diverse but significant requirements. First it must provide a highly reflective surface to meet the previously described physics performance requirements. Second, the laminated PVC structure is self-supporting, even after it is filled with liquid, and therefore must meet specific structural requirements. Third, the extrusion profile is large (66 mm high x 635 mm wide) and yet must meet tight geometric tolerances to facilitate the assembly.

3.1 Reflectance properties

Large gains in the physics performance of the detector and reduction in optical fiber and liquid scintillator costs are realized with even small improvements in the PVC reflectivity. This need for high reflectivity in the 360 nm to 500 nm wavelength range is the primary technical criterion for the PVC. The available rigid PVC compounds such as those used in standard pipe formulations are significantly lower in reflectivity due to less than optimal amounts of TiO_2 and additives that absorb light. Figure 5 shows the large difference in reflectance of a commercially available white PVC and an early NOvA PVC formulation loaded with 15% rutile TiO_2. Although the reflectance of the NOvA formulation is clearly superior, it was difficult to extrude and changes to the formulation were necessary.

In searching for the highest reflectivity, TiO_2 was studied in detail. Our requirements resulted in the need for testing both rutile and anatase forms of TiO_2. Although anatase is not commonly used in PVC, the reflectivity spectrum in the region between 360 nm and 420 nm of anatase is higher than in rutile TiO_2 making it an attractive alternative to rutile. After testing approximately 30 custom PVC formulations, the NOVA-24 formulation was the most promising with rutile TiO_2. Anatase TiO_2 had been tested in other mixtures with great results regarding reflectivity. Since NOVA-24 extruded well with the production die, the same composition was approached for anatase TiO_2 identified as NOVA-27. A graph of the scintillation light spectrum superimposed with the reflectance of NOVA-24 and NOVA-27 extruded PVC

as a function of wavelength is shown in Figure 6(a). The anatase NOVA-27 PVC resin formulation resulted in 14% more light collected (Light Yield) than the equivalent rutile formulation (NOVA-24), as shown in Figure 6(b).

3.2 Mechanical properties

The PVC extrusions form a self-supporting structure. The detector was designed using mechanical properties of typical PVC pipe formulations which guided the mechanical requiremets for the custom formulation. The minimum mechanical properties are listed in Table 1. In addition to the strength and stiffness properties, the PVC must have sufficient impact strength (as a measure of ductility) and exposure to the liquid scintillator should not lower any of these values significantly.

The values in Table 1 refer to the short term (or instantaneous) properties of the PVC. For this structure, the long term structural performance of the PVC is of primary importance. The situation is complicated by the fact that PVC is viscoelastic at room temperature. The most serious implication for the NOvA structure pertains to the structural stability which is a function of the apparent stiffness. The reduction in apparent stiffness over time creates the potential that a structure that is initially stable could become unstable at some future time leading to catastrophic failure. To design against this situation with a quantifiable factor of safety, the time dependent creep modulus is needed at a time equal to the expected lifetime of the structure, which is 20 years. The structural analysis required that the creep modulus (after 20 years) be greater than 517 MPa (75,000 psi) (Table 2). In order to minimize creep related deflections, the design stress in the blocks is kept below 4.8 MPa (700 psi) which is well below the yield and ultimate stresses requirements.

3.3 Geometric Properties

The assembly of extrusions into blocks drives multiple dimensional tolerances. These tolerances include thickness, flatness and straightness of the outside edges (bond two 16-cell extrusions to make one 32-cell module), and the overall flatness (upper and lower extrusion surfaces must attach in the process of block assembly). The NOvA far detector is constructed by bonding the PVC vertical and horizontal extrusions using adhesive. It is essential that the quality of bonding is preserved. The flatness requirement of the PVC extrusions is a key factor to ensure quality adhesive bonding. Only a maximum intra-surface gap of 0.75 mm. (30 mils) across the 240 m^2 bonding surface is allowed. The adhesive is a two-part custom formulation, Plastic Welder 60, devised by Devcon.

Similarly, the extrusion modules must be leak-tight after assembly with the end seals. This drives many tight tolerances on the overall profile such that the manifold and bottom plate mate to two extrusions properly.

The size and complexity of the extrusion along with the high TiO_2 loading made meeting the tolerances very difficult. To meet these strict dimensional tolerances in a production setting required considerable iteration between extrusion process, die design (geometry), PVC formulation, and PVC compounding which were in principle the responsibility of distinct vendors.

4 Need for custom PVC

In order for any PVC compound to be extruded, a number of processing ingredients must be added to the mix. Great care must be taken to ensure that these ingredients have a negligible effect on the reflectivity of the extruded material. Final selection of the components in the formulation was made after almost two years of R&D. In the extruding process, the PVC resin can be subjected to pressures of up to 27.6 MPa (4,000 psi) and temperatures of nearly 194 °C (380 °F) for a significant period of time. The processing

aids (stabilizers and lubricants) are added to the mixture of PVC polymer and TiO_2 to keep molten resin flowing through the die without sticking, burning and decomposing.

Commercially available PVC resins are proprietary and their reflectivity properties are not optimized for NOνA. In order to control all of the ingredients and, in some cases, the brand names of ingredients, we have developed a custom resin formulation. This resin, which meets NOvA requirements for reflectivity, strength, and extrudability, is called NOVA-27 and its composition is listed in Table 3. The net result is a rigid PVC compound with 15% anatase TiO_2 by weight. Mechanical strength tests and light yield tests show no adverse effects due to the use of anatase when exposed to liquid scintillator. NOVA-27 resin produces an extrusion with density equal to 1.49 gm/cc. Given this density and the nominal profile dimensions, the extrusions weigh 15.2 kg/m.

5 Quality Control

Due to the importance of meeting the requirements placed on these extrusions, an elaborate and rigorous QC program was established. This program involved either unique tests or common tests adapted for our purpose. To efficiently carry out these tests, specialized equipment was developed some of which is shown in Figure 7. For QC purposes, a 15 cm (6-inch) cutoff was taken after every 15.5 m (51 foot) extrusion and tested destructively. The cutoffs were subjected to dimensional, impact, pressure, and reflectivity tests. The pressure test (to failure) subjected alternating cells to pressure thereby checking the integrity of the outer wall and web knits. This test was done with a custom pressure tester. The impact test was similar to a drop dart test. Due to the time involved, a custom automated pendulum type impact machine was developed. The reflectivity was tested by measuring the light yield using a HunterLab, UltraScan VIS, reflectometer and a special algorithm. The dimensional tolerances (over 150 dimensions) were checked using an OGP, SmartScope Flash 500 optical measuring device which also was automated. In addition, each extrusion was subjected to a vacuum test (non-destructive) to confirm the web integrity

over the length of the extrusion. This custom device was also automated to save time. An additional cutoff was taken daily and sent to ANL where samples were made for tensile testing of the bulk material and the outer wall and web knits. All of the data was uploaded daily to a project database.

6 Discussion / Lessons learned

6.1 Extrusion

The first attempts in PVC formulation were done with the intent to make PVC compounds with only a few components and large amounts of rutile TiO_2. These products tended to be dry and did not extrude well in industrial conditions even when using a small and less complex die with just 4 cells. It was found that it was necessary to include most of the components found in a typical rigid PVC formula. Therefore the approach became one that attempted to minimize absorption in the components. Shintech SE950EG was chosen for its superior clarity. As stabilizers both Advastab TM-181 (20% monomethyl tin) and TM-182 (80% monomethyl tin) were tested. Although TM-182 yielded an extrusion with slightly higher reflectivity than TM-181, it did not protect over time as well from degradation during the extrusion process. Several rutile and anatase TiO_2 products were tested. Kronos 1000 anatase TiO_2 was selected because it yielded the highest reflectivity. The calcium stearate, paraffin wax, oxidized polyethylene, glycerol monostearate, acrylic impact modifier, and processing aid underwent a few product changes from the early R&D process to the final extrusion production mostly due to changes in availability. Nonetheless once the components were fully tested, the formulation was unchanged.

During R&D, it was important to test the PVC formulation under extrusion conditions similar to those of the final production. The problem was not the reliability of the reflectivity results. Rather, it was found that a short extrusion run using a small extruder with a small die did not expose the PVC compound to the extrusion conditions encountered in an extrusion test utilizing a large die, in particular a very wide die like the one needed for the NOvA extrusions. Because results varied under different extrusion conditions,

it was necessary to have the NOvA die and the final extruding line setup to learn about the actual stability properties of the PVC compound formulated and the mechanical properties of the extrusions prepared. NOVA-27 PVC while meeting all of the performance requirements set out above was a difficult compound to prepare and to extrude.

6.2 Mechanical properties

The preparation of the NOVA-27 PVC required close attention. The untreated anatase TiO_2 added variations to the final PVC compound which affected the extrusion process and the final mechanical properties of the extrusion. Initial developments with rutile TiO_2 provided excellent ductility in standard tensile tests and high impact strength. The switch to anatase TiO_2 initially brought with it serious reductions in ductility and impact strength. After considerable efforts, ductility comparable to that originally obtained with rutile was achieved by improving the dispersion of the anatase. Over the course of production, variations in ductility were observed between the bottoms and tops of the extrusion.

From a QC point of view, drop dart impact tests served as a good quick on-site leading indicator of ductility issues although tensile testing was used as the final decision of ductility.

Time-temperature superposition tests were used as a method of estimating the long term creep of the NOvA formulations. The creep properties were found to be relatively independent of formulation. Predictions based on accelerated creep tests showed good conservatism when compared to long term studies. For PVC, an effect referred to as “physical aging” results in a lower observed creep rate. To explain this, we defined an aging time, increasing t_e , which is the time elapsed from the time that the material is extruded to the time of initial load application in a creep situation. It has been shown for PVC that increasing t_e by a factor of 10 decreases the creep rate by a factor of 10 and shifts curve along the time scale to the right [2,3]. This effect was observed in the NOvA materials. The creep tests revealed that for our material, stresses ranging from 2-4.8 MPa (300-700 psi) showed no significant stress

dependence and are consistent with the claim of linear viscoelastic behavior below the design stress of 5.0 MPa (730psi).

Due to the complex shape of the die and high number of internal cells, the extrusion profile has a very high number of knit-lines where material passes over a die support and rejoins shortly before exiting the die. There are 77 knit locations in the profile (Figure 8). While most performed well, the central knits in the webs were of reduced strength and ductility compared to the bulk material. This reduction is generally more pronounced in the central webs (3-13) while the outermost webs (1,2,14, 15) reveal no serious reduction. A clear difference between the break at elongation is seen between the outer web (1) and the inner webs (4,8,10) as shown in Figure 9 . Inspections of the failure surfaces revealed that the web 1 samples failed in the bulk material. That is, the knit line was as strong as the bulk material. The exact opposite is true for the inner webs which all failed clearly at the knit line. The ultimate tensile strength (UTS) of web knits 1 is consistent with the bulk material UTS. Similarly, one can see that elongations well above 20% are generally expected. Figure 10 shows a comparison of the UTS and elongation at break during the web knit test for webs 1 and 8. This reveals that the web 1 knits behave similar to the bulk material whereas the web 8 knits have reduced strength and markedly reduced elongation. The failure strain of the worst knits was found to be on the order of 2% which is greater than the design strain over the twenty year lifetime. Preliminary data from creep rupture tests of the knitlines (applying a fixed load and recording the time of failure) indicated that these worst webs have sufficient creep lifetime. More details of this work is reported in [4].

6.3 Geometry

The assembly of the modules and blocks required relatively tight tolerances. Achieving these was problematic particularly due to the difficulty of extruding our unique formulation. The biggest challenges were maintaining thick enough webs without creating an imbalance in the overall profile thickness. There

was also trouble in maintaining the internal cell radii although it could be expected due to the lack of sizing for internal cells. While we had to accept dimensions in some cases that were different from those originally planned, we did find the final dimensions to be relatively repeatable which was our primary goal. In our early production experience, we found that we had to open up several tolerances such as the web location in order to not unnecessarily reject otherwise excellent extrusions. Our choice was to make a modification to the auxiliary matching parts as needed.

7 Summary/conclusion

A complex PVC extrusion was developed and produced to serve as the main component of a large, self-supporting structure, filled with liquid scintillator, and designed to stand for 20 years. The high TiO2 loading presented considerable difficulties in extruding, effectively narrowing the operating window available to the machine operators. The development stage spanned a 4 year period, where a number of PVC formulations and extruding techniques were tested, leading to the optimal PVC formulation, tooling and extruder configuration. A successful around-the-clock production ensued, spanning a period of 3 years and using over 7 million kilogram (15 million pounds) of PVC powder. Approximately 22,000 15.5 meter-long extrusions for the Far Detector were produced, along with extrusions for the Near Detector and an equal number of 6 inch cutoffs for quality control evaluation.

8 Acknowledgements

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9 References

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Table 1 Minimum mechanical properties of the NOvA PVC compound

Material Property	Value	Test Method
Modulus of Elasticity (Instantaneous, t=0)	3.1 GPa (450,000 psi)	ASTM D638
0.2 % Offset Yield	27.6 MPa (4000 psi)	ASTM D638
Ultimate tensile Stress	37.9 MPa (5500 psi)	ASTM D638
Creep Modulus	See Table (2)	ASTM D2990

Table 2 Minimum values of time dependent (Creep) Modulus

Time [Years]	Modulus [GPa/ (psi)]
0.5	1.81 / (262,631)
4	1.0 / (145,544)
10	0.69 / (100,713)
20	0.50 / (72,046)

Table 3 Composition of N-27 rigid PVC compound.

INGREDIENT	COMMERCIAL BRAND NAME	PARTS PER HUNDRED	PER CENT
PVC	Shintech SE950EG (high reflectivity)	100	77.5%
Tin stabilizer	Rohm & Haas Advastab TM-181 20% monomethyl tin	2.5	1.9%
Titanium dioxide anatase	Kronos 1000 anatase titanium dioxide	19	14.7%
Calcium stearate	Ferro 15F calcium stearate	0.8	0.6%
Paraffin wax	Ferro 165 paraffin wax	1.1	0.9%
Oxidized polyethylene	Ferro Petrac 215 oxidized polyethylene	0.2	0.2%
Glycerol monostearate	Rohm & Haas F1005 glycerol monostearate	0.3	0.2%
Acrylic impact modifier	Arkema Durastrength 200 Acrylic impact modifier	4	3.1%
Processing aid	Rohm & Haas Paraloid K120N processing aid	1	0.8%
	Total	129	100%

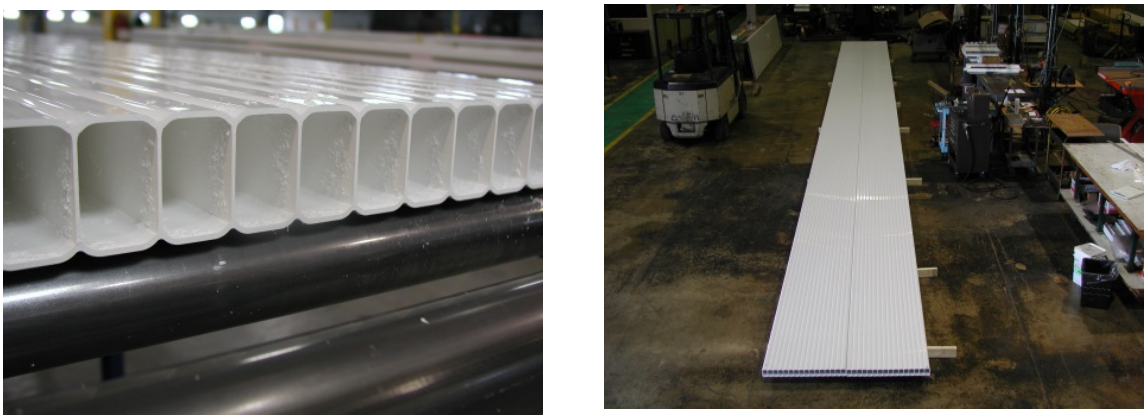

Figure 1 (a) Closeup of 16-cell PVC extrusion, freshly cut. (b) Two full-size 16-cell extrusions 15.5 m (51 feet) long placed side-by-side.

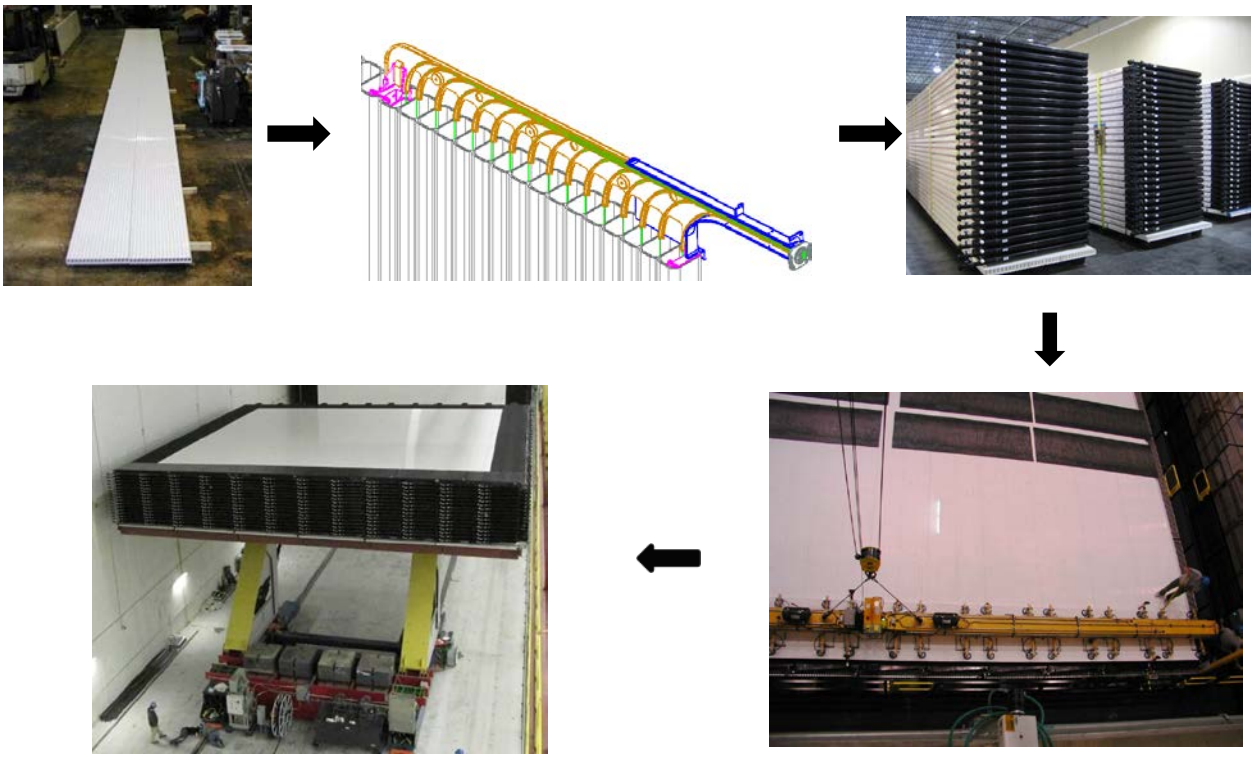

Figure 2 Extrusions are glued edgewise to form modules which are then arranged side by side to form a 15.5 m (51 foot) square plane.

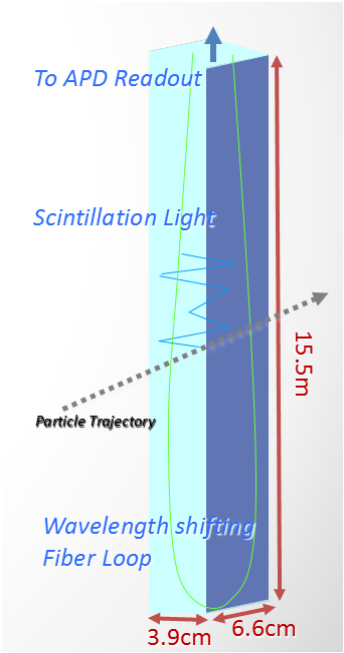

Figure 3 Radiation particles passing through the scintillating liquid within a cell produces light which reflects off the PVC walls multiple times until being captured by a “wavelength-shifting” fiber optic loop. Light within the fiber optic light travels the length of the extrusion and is detected by an “APD” photo-detector.

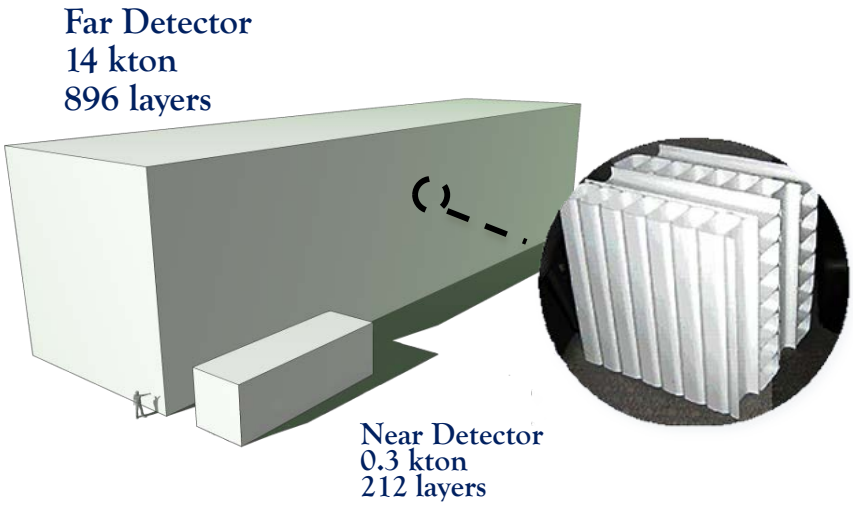

Figure 4 Schematic illustrating the size of the two NOvA detectors and the alternating orientation of the PVC extrusions. Note the person depicted in the figure to provide a reference scale.

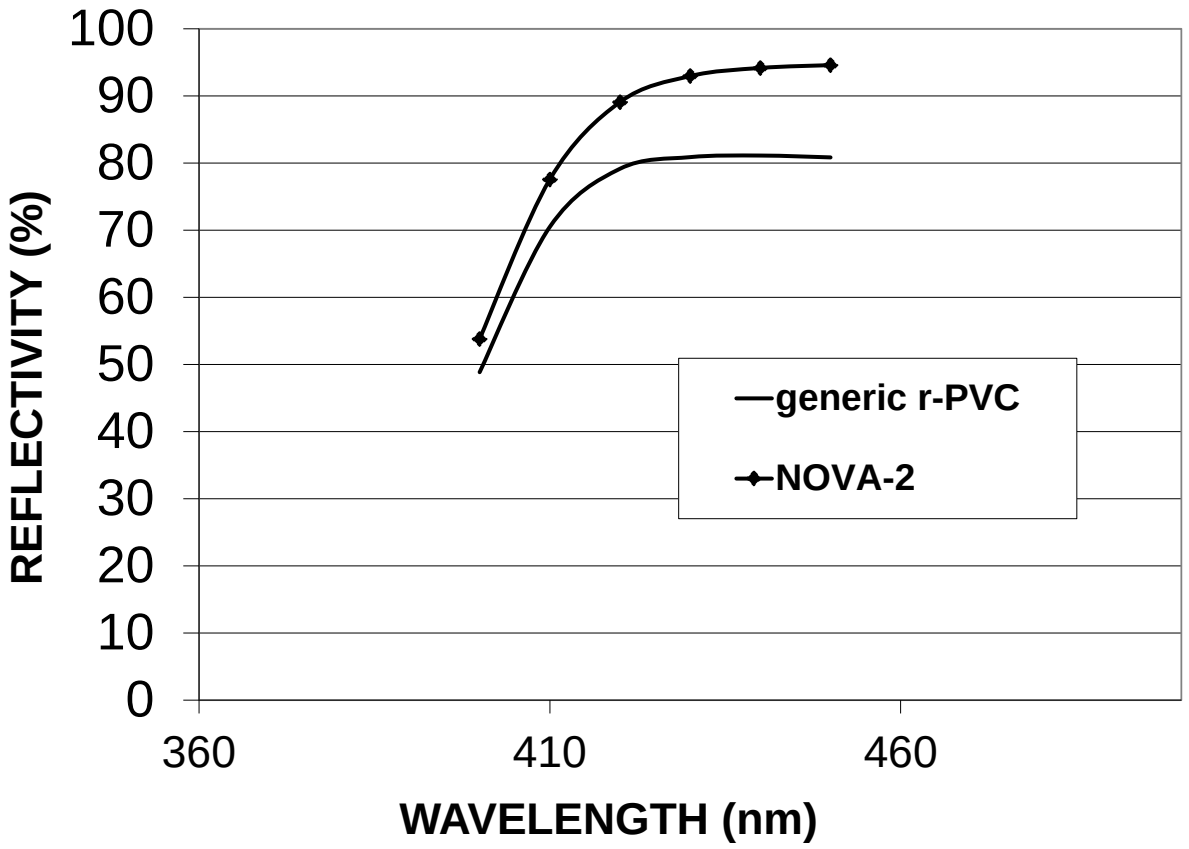

Figure 5 Reflectance of commercially available white PVC (generic curve) is compared to the reflectance of an early NOvA formulation loaded with 15% rutile TiO_2 (NOVA curve).

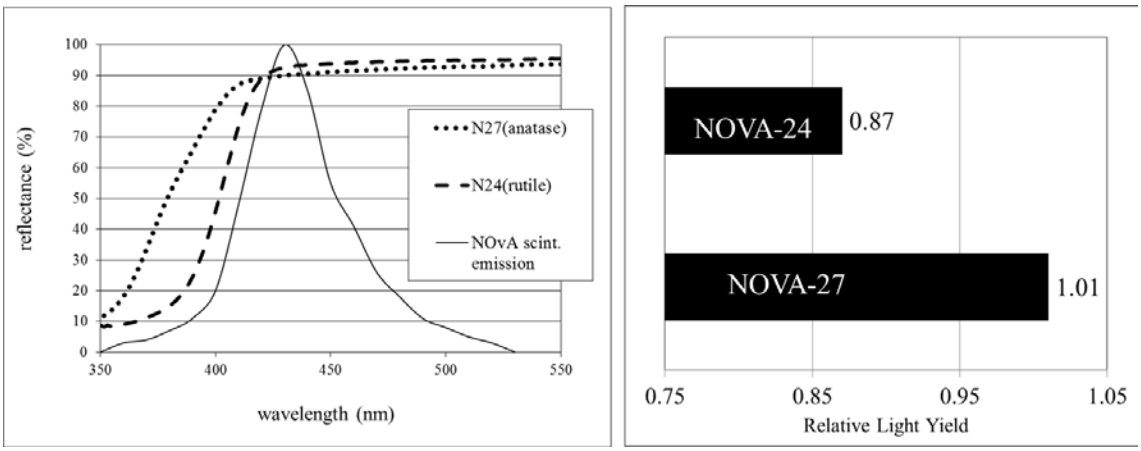

Figure 6 (a) Scintillator light spectrum (solid black) is superimposed on a reflectance graph of extruded PVC made with NOVA-24 (15% rutile) shown in dashed line and NOVA-27 (15% anatase) shown in the dotted line. (b) The total light collected from a scintillator-filled extrusion made with NOVA-24 (lower bar) and from a scintillator-filled extrusion made with NOVA-27 (anatase).

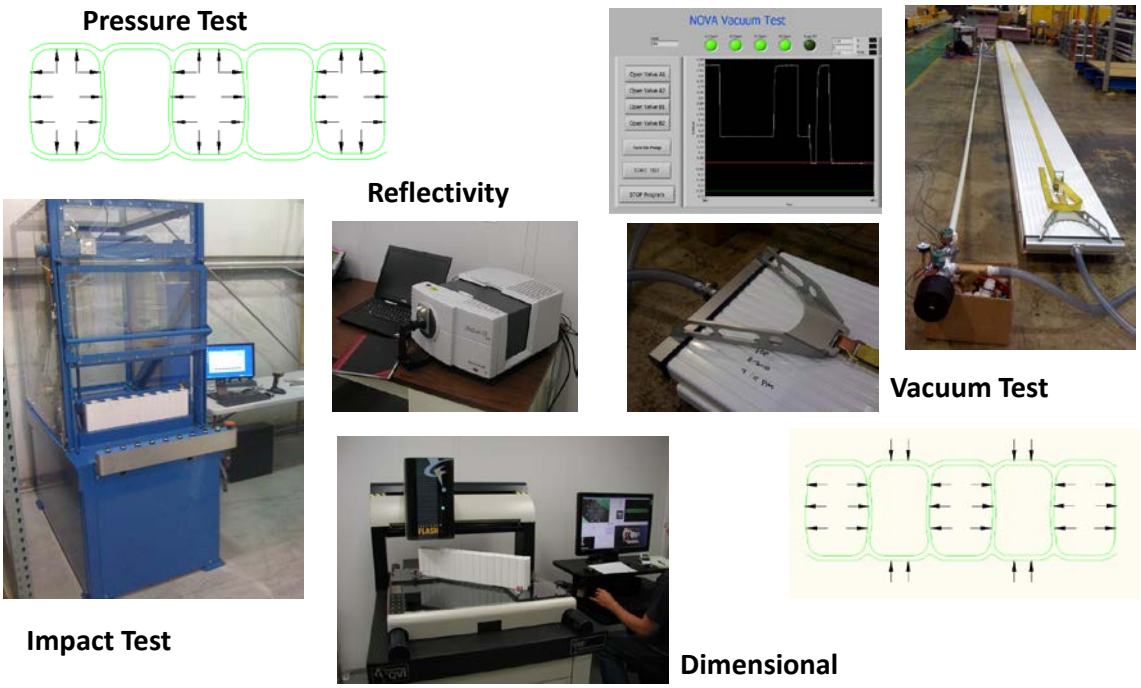

Figure 7 Some of the unique QC tests and equipment designed for these extrusions.

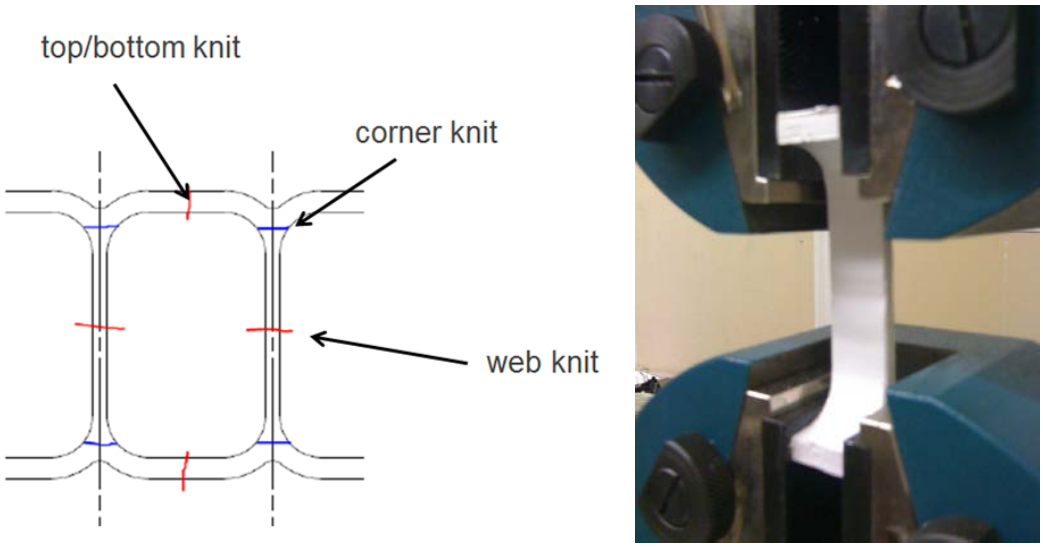

Figure 8 Locations of knit lines in a single cell and the web knit tensile test specimen and set-up.

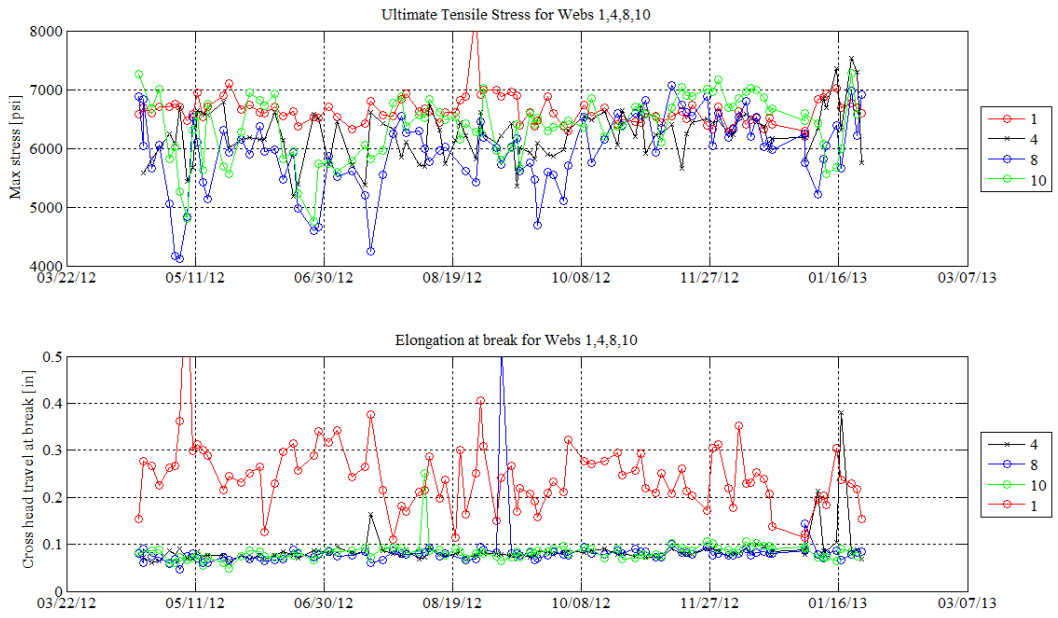

Figure 9 Ultimate tensile tress and elongation for webs taken from QC testing.

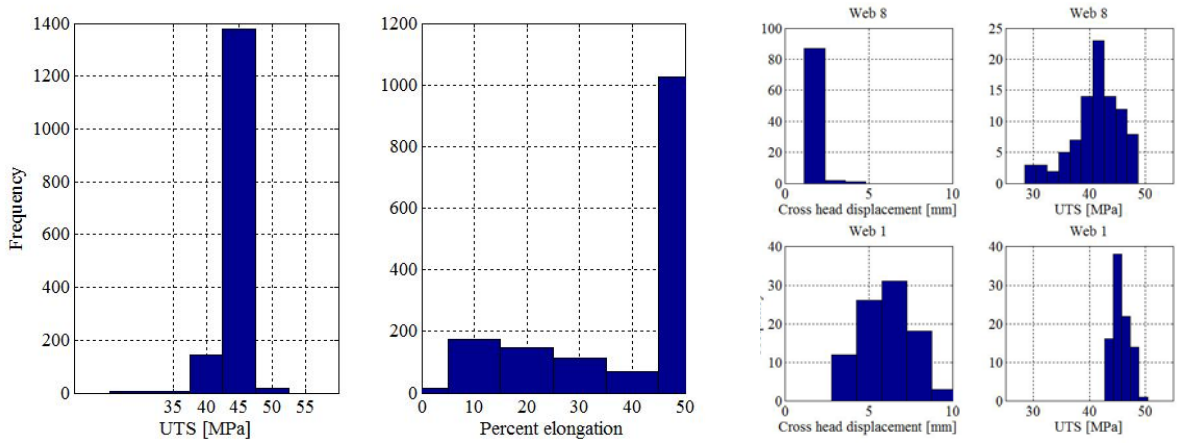

Figure 10 Left plot: Histogram for UTS and percent elongation for standard tensile samples made from start of production until Jan. 2012. Right plot: Histogram showing difference in elongation and ultimate tensile strength of central (web8) and outer (web 1) webs.