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DRAFT: EVALUATION OF MECHANICAL PROPERTIES AT THE KNIT LINE INTERFACE IN A COMPLEX MULTI-CELL PVC EXTRUSION

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ABSTRACT

In order to form internal cells in PVC extrusions, the die requires an insert or internal member that the material flows past. These inserts are supported to the outer die structure by so-called spiders that pass through the extrusion wall. The extruded material must separate and the recombine as it passes over the spider. The material must then form a bond at this interface shortly before exiting the die. These interfaces are referred to as knit-lines. In a recent project involving a large and complex PVC extrusion, difficulty was encountered in developing these knit lines within the webs of the extrusions. Upon visual inspection, these interfaces appeared to be without bond over portions of the cross section. However, mechanical testing in the worst knits revealed that bonding had occurred with the knit providing 85% or the bulk material strength although without support any significant ductility. At the same time, knits at different parts of the extrusions showed ductility comparable with the base material. Altering the process variables showed a means for improvement in the webs but this was limited by other constraints. While these knits were sufficiently strong for short duration loading, the real question was the time dependent strength such as creep rupture over long times and how the behavior of the knit compared with the bulk material. In this work we describe the character of the knitlines and the resulting mechanical properties. We then discuss the preliminary results of accelerated creep rupture tests which indicate that the creep rupture behavior of the partially interfaces follow a time temperature superposition relationship allowing accelerated prediction of the knit-line rupture. We propose a model for use in analyzing the knit-line bonded in such fashion.

NOMENCLATURE

UTS	Ultimate Tensile Strength
PVC	Polyvinyl Chloride
ASTM	American Society for Testing and Materials

INTRODUCTION

A large complex extrusion multicell PVC extrusion profile has been developed for use as a physics detector [1]. The extrusion provides the basis of detector modules which are assembled form a large self supporting structure. Due to the physics purpose of the structure, an additional and driving requirement for the extrusions is that that the interior surface have high reflectivity over a particular wavelength of light. This is in addition to structural requirements and the requirement that the material be extrudable in the profile required. In the extrusion die industry, shapes such as this are referred to as profiles [2] and are generally distinguished from pipes with a main difference being that profiles are not typically required to fulfil structural requirements. Due to these unique requirements, a custom PVC formulation was developed which was dominated by a high loading of TiO_2. The material need the strength of pipe formulations, with the processing ability of a profile formulation while being highly reflective (and not absorptive) in a select wavelength of light. The components of a the final formulation, so-called NOvA-27, are listed in Table 1. This formulation develops a bulk PVC material

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FIGURE 1. EXTRUSION PROFILE AND THE WEB KNIT-LINE SAMPLE AS CUT FROM THE EXTRUSION.

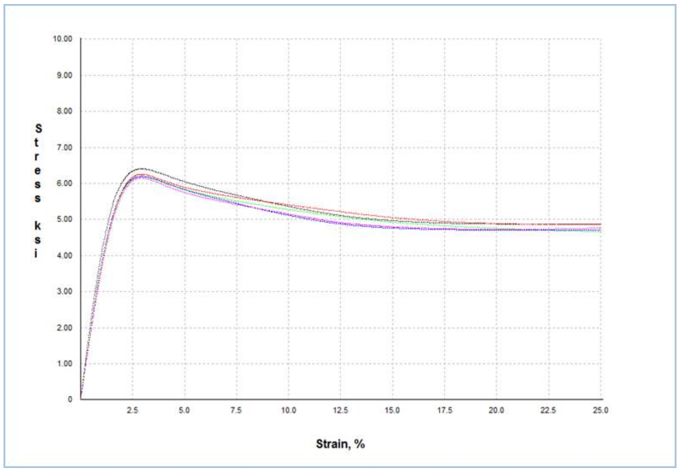


FIGURE 2. STRESS STRAIN CURVE FOR THE BULK NOvA-27 MATERIAL

with sufficient mechanical properties and substantial ductility as evidenced by the stress strain curve in Fig. 2.

The combination of the large and complex profile compared with high TiO_2 loading presented a difficult challenge in developing parts that also met relatively tight geometric constraints. For example, the processing parameters that were most favorable to meeting the geometry requirements were not the optimal parameters for the other requirements.

The PVC extrusion as shown in Fig. 1 contains 16 internal cells. These internal cells are formed with inserts in the die which must be held and supported from the outer surface of the die. These supports are often referred to as spiders. As the material flows through the die, it must separate and then rejoin as it flows over these spiders. The material rejoins at an interface in the material often referred to as a weld or knit line. The quality of this interface can depend on numerous factors including the die design, the PVC formulation, and the processing conditions. For example, the amount of internal or external influences knitting [3]. Features of the die such as compression and spider design have an effect [4] but optimizing this can be difficult in complex dies. Increasing temperature has a positive effect improving the knitting but there is a limit on the temperature before the PVC start to become degraded [**?**]. Huang [4] also notes that the mechanical properties are frequently lower in the knit lines. Broutman performed impact analysis of a failed water pipe and found no weakness compared to the bulk material [5].

The complexity of the NOvA extrusion profile dictates a large number of knit locations. Figure 4 shows a single cell of the extrusion indicating the location of the insert spiders and resulting knit locations. The figure shows that there are eight knit lines associated with each cell (noting that each web is shared by adjacent cells). The knit lines are grouped into three types based on their location in the cell. The knits are identified as web, side, and corner knits corresponding to the location. With respect to the flow direction through the die, the insert supports are located just prior to the exit of the die. Along the flow direction, there are only four supports at any cross section. That is, the four corner supports from which corner knits develop are located immediately prior to the die exit. As you move toward the inlet of the die, the inserts are then supported from four more centrally located supports from which the side and web knits are developed.

The extrusions in general were subjected to a large number of QC tests including a pressure test to confirm the quality of the knits. A passing extrusion was considered to be one which would maintain greater than 200psi (1.38 MPa)in a short term pressure test which greatly exceeded the maximum design pressure of 19psi (0.13 MPa). While extrusions were able to pass the pressure test, the failure was always at a knit line. In the initial development of the die, the corner knits were found to be the weakest. A considerable improvement to these knits was made by reducing the length of the corner supports at the exit of the die such that the material had a increased travel distance within the die while recombining to form the knit interface. The side knits were generally found to behave similar to the bulk material in both strength and elongation measurements. This left the web knits and specifically those in the more central region of the extrusion as the weakest link. Considerable effort went into trying to improve and characterize the quality of the knits. That is the subject of this paper.

WEB KNITS

The pressure test was done on 6 in (153 mm) samples and was destructive such that the pressure was increased until failure occurred. While the NOvA extrusions profiles were passing

TABLE 1. COMPOSITION OF N-27 RIGID PVC COMPOUND.

Ingredient	Commercial Brand Name	Parts per hundred 2	Percentage
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	19	14.7%
Calcium stearate	Ferro 15F	0.8	0.6%
Paraffin wax	Ferro 165	1.1	0.9%
Oxidized polyethylene	Ferro Petrac 215	0.2	0.2%
Glycerol monostearate	Rohm & Haas F1005	0.3	0.2%
Acrylic impact modifier	Arkema Durastrength 200	4	3.1%
Processing aid	Rohm & Haas Paraloid K120N	1	0.8%
Total		129	100%

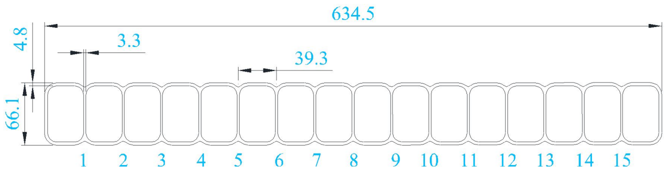


FIGURE 3. EXTRUSION DIMENSIONS AND WEB NUMBERS.

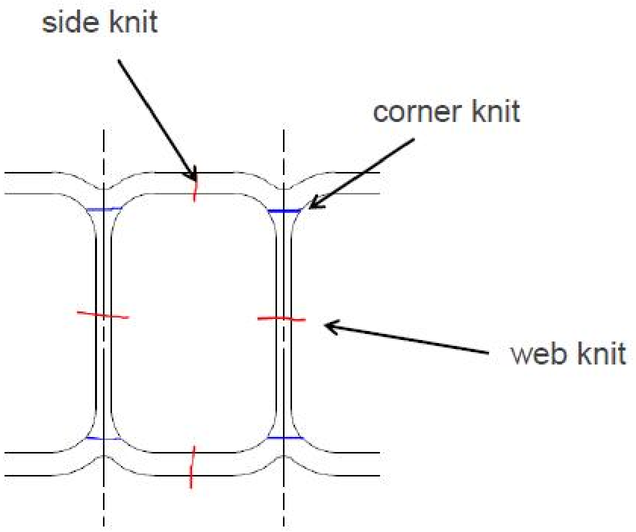


FIGURE 4. LOCATION AND IDENTIFICATION OF KNIT LINES IN CELLS 2-15 (OUTER WALLS OF CELLS 1,16 DO NOT HAVE WEB KNIT).

the (intentionally destructive) pressure tests, investigation of the failure surface at the location of the web knit line gave the appearance of incomplete or partial knitting. A failure surface is shown in Fig. 5. This type of surface was typical of the central webs particularly 7-9 as identified in Fig. 3. At first appearance, the smooth interior surface appears to be a void or crack interface. This is not the case as will be discussed below. In order to characterize the quality of the these webs as well as develop a more objective criteria to evaluate these interfaces, a web testing procedure was developed and used to evaluate the webs over a variety of production conditions.

Test Procedure

The web test specimens were cut directly from 1/2 inch (12.7mm) wide cross sections of extrusions (Fig. 1 leaving the shape shown in Fig. 8 . Tensile testing was performed on the specimen using a standard universal tensile test machine. The samples are placed in the grips just above the start of the corner radii such that the samples behave somewhat like dogbones leaving the web area under the highest stress (Fig. 6). The main goals of these tests was to characterize the strength and ductility of the knit line interface. For these goals and based on the test results, the samples do not add any systematic failure. That is, the failures tend to occur due to necking of the thinnest cross sections and or at the knits which are expected to be weaker than base material. The samples were run with the cross head speed set at 0.5 in/min (0.21 mm/s) . A few tests were run at 0.05 in/min (0.021 mm/s) to examine the sensitivity to rate effects with no

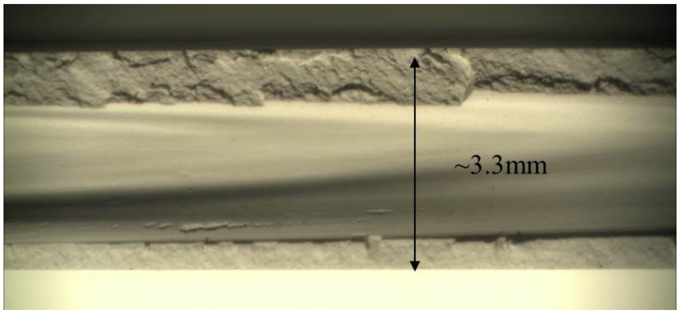


FIGURE 5. FAILURE SURFACE ALONG WEB KNIT LINE OF AN EXTRUSION EARLY IN PRODUCTION.

difference in results. An extensometer small enough for these samples was not available nor deemed necessary for the stated goal and so the cross head displacement is used as a measure of elongation. It is noted then that references to elongation data in this paper refer to the crosshead position measured during the test. Due to crosshead compliance and occasional slipping in the grips, this value needs to be interpreted lightly when trying to equate to a strain value. In the discussion of the test results, classification of ductile/brittle failure while consistent with the gross elongation ranges was determined by visual inspection and the observance of necking. The gage length is conservatively estimated at approximately 1.8 +/-0.2 inches (45.7 +/- 5 mm). The samples were cut on a band saw with a relatively fine blade. Nonetheless, saw marks remain on the thinnest faces of the specimens and no effort was made to remove these. Since the samples failed consistently at the knitline and in many cases with good ductility, it is not believed that these saw marks were the cause of premature brittle failure (or random failure location would occur). However, in very ductile high extension with considerable necking, the failure generally developed starting at one of these edges and propagated slowly as a tear across the section. It is likely that further elongation in these cases was limited by the rough outer surface. This test procedure was developed with the intent of being a frequent QC test during long term production of the extrusions. As such, it was desirable to use minimal effort on specimen preparation.

The samples are pulled in the axial direction and the load and position of the crosshead are measured. As noted above, the position of the cross head is representative of the specimen elongation and strain but the absolute values should not be interpreted so closely due to effects such as slipping in the grips. Typical load versus displacement plots for the specimen are shown in Fig. 11. Three distinct types of curves are observed and are identified as: 1.) straight line curves indicating failure with essentially zero inelastic deformation (brittle), 2.) curves showing reduction in force and minor inelastic elongation at failure , and 3.) curves showing significant drop in load and considerable elongation (ductile). We use the definitions in this paper and not that different definitions for types 1-3 failure may be encountered in the literature. Type 2 responses indicate some necking and inelastic elongation as evidenced by the load drop indicating strength comparable to the bulk material . These three types are illustrated in Fig. 11. The bulk material always exhibits type 3 behavior.

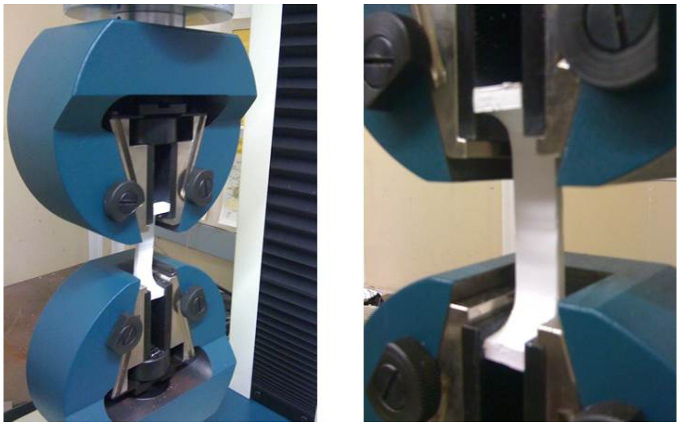


FIGURE 6. SPECIMEN IN GRIPS SHOWING APPROXIMATE GAGE LENGTH.

Extrusion Sample Description and Processing Conditions

In an effort to characterize and potentially improve the web knitlines, a series tests were run over several days with representative extrusion samples cut and made into web test specimen for evaluation. These samples were identified with letters and the main test conditions are listed in Table 2. The test conditions included two different sets of screws used in the extruder identified as 'old' and 'new'. The difference between the screws was that the 'new' screw provided additional mixing and frictional heat. In addition, extrusion temperatures were varied in order to increase the melt temperature and tests were conducted with and without a breaker plate to evaluate the effects of additional mixing and dispersion. The tests and analysis pertain only to the web knit lines as identified in Fig. 4.

Results of Web Tensile Testing

In testing of all 15 webs from a single extrusion, load/displacement curves of the various types described in Fig. 11 are observed in some combination. The the outermost webs (1,2,14, 15) reveal no serious reduction of strength or ductility at the knit lines and the failure generally occurs in the bulk material. In the more central webs (3-13), failure almost always occurs at the knitline with either types 1 or a type 2 failure.

The following observations were made when viewing all of the data:

TABLE 2. Extrusion Sample Descriptions

Sample	$T_{m}elt[^{\cdot}C]$	Breaker Plate	Screw
C	194.4	no	new
E	189.4	yes	new
F	194.4	yes	new
H	189.4	yes	new
J	185.6	no	old

1. 65 specimens were tested: 53 of the specimens failed at the knit.
2. The lowest failure occurred at 5400 psi (37.2 MPa) and most failed at stresses comparable to the bulk material stress ($>$ 6000 psi [41.4 MPa]). On average, the failure stress was the same for all samples. See Figure 5.
3. Samples C and F exhibited better ductility that sample J,H, and E
    - (a) Samples C and F had 10 of 30 specimens fail outside the knit line and 16 of 30 fail in ductile fashion. This compares to samples J, H, E of which only 2/25 failed outside the knitline and 7/25 failed in a ductile manner.
    - (b) Ductility across the knitline is generally poorer in the interior webs. For samples C and F, cells 5-11 failed with limited ductility (type 2)interior cells . For J,H, and E samples, cells 3-13 failed brittle and exhibited the much more brittle type 1 failure.
    - (c) A view at Figure 6 shows also that the brittle failures of samples C and F are generally the more desirable type 2 as compared to the brittle failures of samples J,H, and E are the very brittle type 1. Further it can be seen that the brittle failures of samples C and F while having minimal elongation appear to approach the yield stress of the material just before failure as evidenced by the minor load reduction. For the brittle failures of samples J, H, and E, there is not only zero perceptible inelastic elongation but these samples fail below the yield stress of the material.

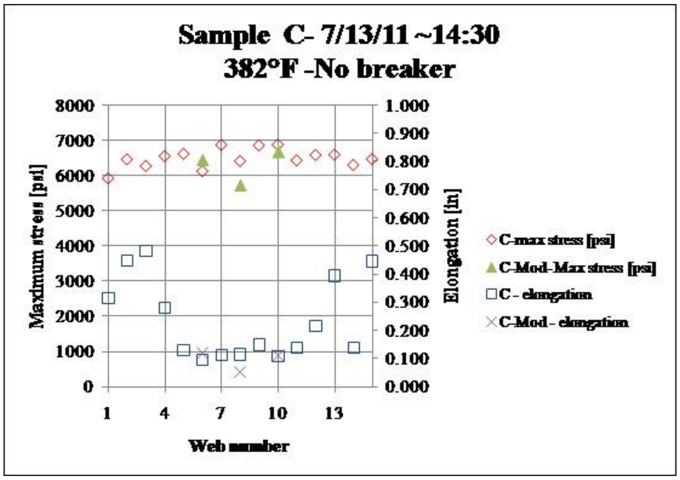


FIGURE 7. PLOT OF ULTIMATE TENSILE STRESS AND MAXIMUM ELONGATION FOR SAMPLE C AND MODIFIED C SAMPLES.

Figure 7 shows the ultimate stress and the cross head displacement for the specimens from test sample C. The ultimate tensile strength (UTS) is near that of the base material and always greater than 85% the ultimate strength. More pronounced is the difference in ductility when failure occurs. The elongation at break expressed as crosshead travel. A clear difference between the break at elongation is seen between the outer web (1-3,13-15) and the inner webs (4-12). 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. While the strain is not directly measured, this strain is estimated to be about 2-3%. This is consistent with an optical strain measurement shown just before failure in Fig. 17.

As further comparison to the bulk PVC properties, Fig. 9 is a histogram of the UTS and failure elongation for the standard tensile samples taken from the tops and bottoms of the extrusions (with no transverse knit lines). The test method and tensile specimens are standard type 1 specimens as defined in ASTM D638 and are performed in the extrusion direction or perpendicular to the direction of the web specimens. In the context of these web tests, the bulk material was found to behave similarly in the transverse direction. It is also noted for that data that the test is stopped at 50% elongation (2in [50.8mm] gage length) The 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 **??** 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.

Test Of Flat Knit-Line Surface

Many of the brittle failures at the knit-line reveal a two zone surface appearance with a smooth inner area and a rougher out area (Fig. 5. In many cases, the inner smooth area is so smooth as to give the appearance as a complete lack of adhesion (see for example web 6 in Fig. 13). Two questions arise: 1.)is this interface so weakly bonded that it behaves as a pre-existing void that can lead to crack propagation and 2.) how does the strength

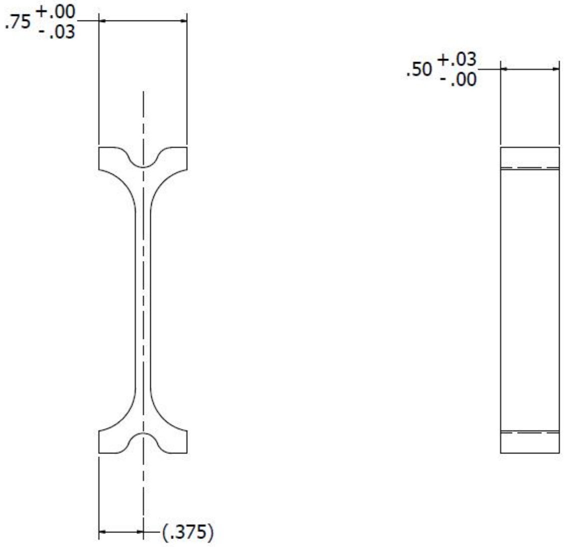


FIGURE 8. GEOMETRY OF WEB KNIT TEST SECTIONS (DIMENSION IN INCHES).

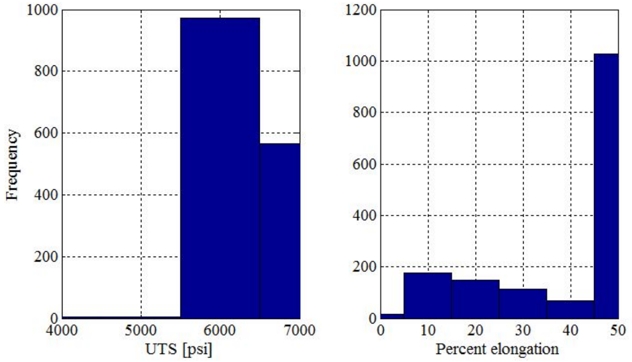


FIGURE 9. HISTOGRAM FOR UTS AND PERCENT ELONGATION FOR STANDARD TENSILE SAMPLES.NOTE: TEST STOPPED AT 50% IF FAILURE HAS NOT OCCURRED.

across this interface compare with the base material. To answer these questions, several specimens from the C sample were modified by removing material from the outer surfaces on both sides of the specimen in the area of the knit line. The specimen is shown in Fig. 14. Figure 14 also shows the resulting fracture surface of two of the samples. The upper fracture surface (C2-Mod-8) shown in the figure shows a 100% smooth knit interface with no evidence of adhesion. However, the knitline did not fail until 5600 psi. While this is lower than the bulk material, it clearly confirms that the smooth surface forms a significant bond. The sample failed without inelastic deformation indicating extremely limited ductility across this type of interface.

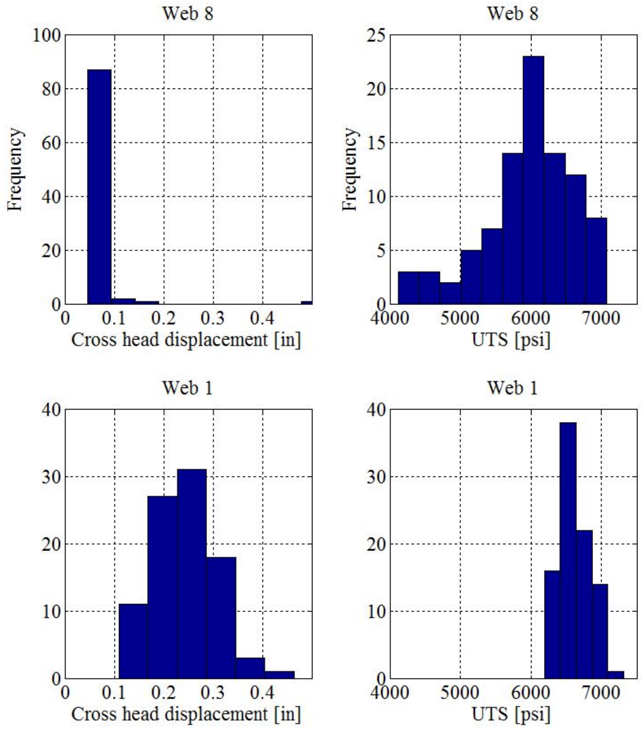


FIGURE 10. HISTOGRAM SH0WING DIFFERENCE IN ELONGATION AND ULTIMATE TENSILE STRENGTH OF CENTRAL (WEB 8) AND OUTER (WEB 1) WEBS.

The lower fracture surface on lowesample (C2-Mod-6) reveals only two distinct regions and does not have the typical symmetry associated with the full sections. This is a product of how the reduced section was cut and indicates that revealing a smooth inner surface is not a consequence of the test but inherent to the web material. This sample failed in a ductile manner and at a sufficiently high stress. These tests cleary reveal that the smooth surface does not indicate an apparent lack of adhesion and that significant strength is transmitted across these smooth surfaces. It is important to note that these surfaces are not able to sustain the full bulk material yield stress and are not able to support large deformation associated with plastic flow of material.

LONG TERM STRENGTH

The web knit test results reflect short time (load applied over minutes) tensile tests and one cannot conclude that the knits would survive to the same level of creep strain under long loading times. A preliminary study of the creep rupture times for the web knits was undertaken.

Creep rupture tests involve applying a fixed load and recording the time of failure. It is expected that the failure stress plotted versus log time is linear (with a conservative departure from lin-

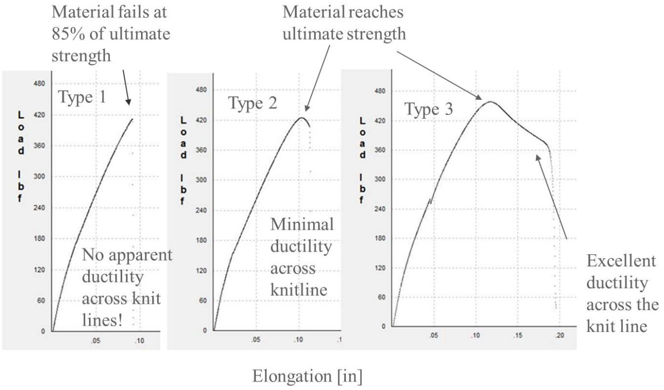
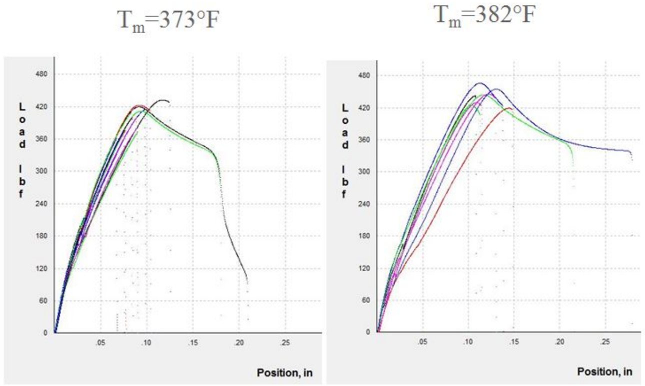


FIGURE 11. CLASSIFICATION OF TENSILE TEST BEHAVIOR ACROSS WEB KNIT LINE. WEB TESTS EXHIBITED ONE OF THE THREE TYPES OF FAILURE ABOVE. TYPE 2 AND 3 ARE ACCEPTABLE. TYPE 1 IS QUESTIONABLE.



Inner webs –type 1
Outer webs –type 2

Inner webs –type 2
Outer webs –type 3

FIGURE 12. COMPARISON OF KNIT LINE DUCTILITY AS A FUNCTION OF TEMPERATURE COMPARISON.

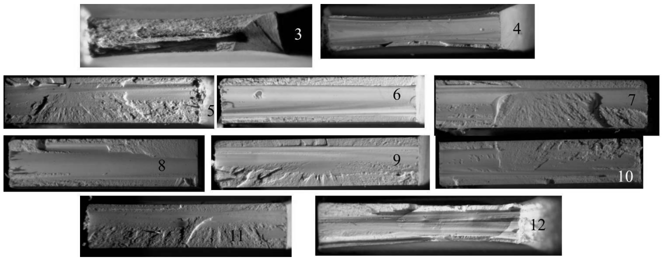


FIGURE 13. FRACTURE SURFACES FROM SELECTED WEB TESTS. WEB NUMBER INDICATED IN PHOTO. WEBS NOT SHOWN COMPLETED TEST WITHOUT COMPLETE FAILURE.

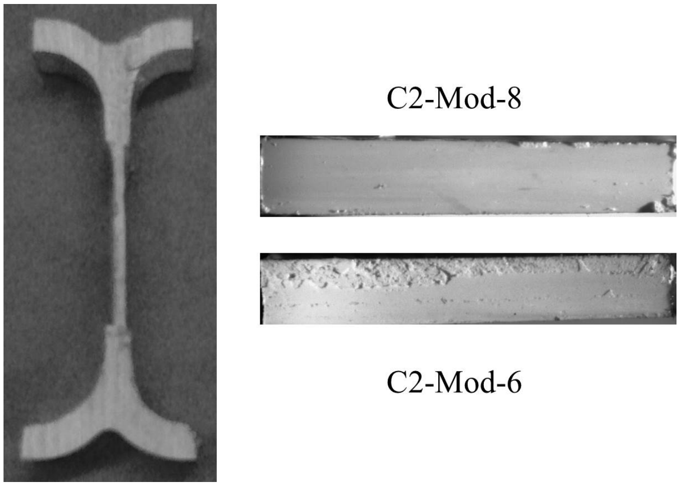


FIGURE 14. Modified sample used to assess mechanical properties of a smooth knit-line surface and fracture surfaces from two of these.

earity at low stresses). Using this linearity, longer time creep rupture can be extrapolated. Initial creep rupture testing was done at ANL using the Universal testing machine for various stresses such that failure was recorded in times on the order of seconds and days. For much longer time, a fixture was constructed at FNAL and two tests were run at lower stress and for much longer times. Both tests were performed at nominal room temperature using samples cut from web 7 which are considered to be among the worst. The results are shown in Fig. 16 and labeled as ANL-Tinius Olsen and FNAL-fixed weight respectively. Due the time required to run these tests, it was decided to perform these tests at elevated temperature with the hopes of gaining predictive capability and further to confirm that the failure mechanism was similar. Due to the need for special equipment, this task was contracted to IIT in Chicago as phase 1. Preliminary creep rupture tests were performed at 60 C to evaluate the process. These results are also shown in Fig. 16 and labeled as at 60 C. As is seen in the figure, the data is consistent with the expected shift in log time due to the higher temperature consistent with an Arrhenius relationship.

DISCUSSION

Figure 15 shows the types of failure surfaces encountered in the web tests. The letters in the figure correspond to the following description:

A. Complete ductile failure away from knit line.
B. Ductile failure at the knit line.
C,F. Brittle failures revealing some areas of relative stronger bonding.
D. Brittle failure with extremely smooth interface. These surfaces are consistent with the least ductile and weakest

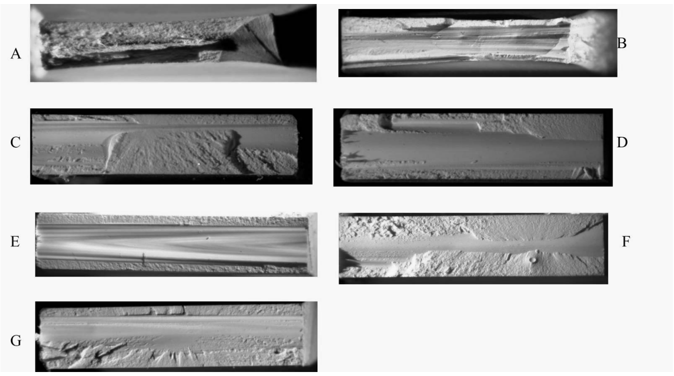


FIGURE 15. IMAGES OF KNIT CROSS SECTIONS.

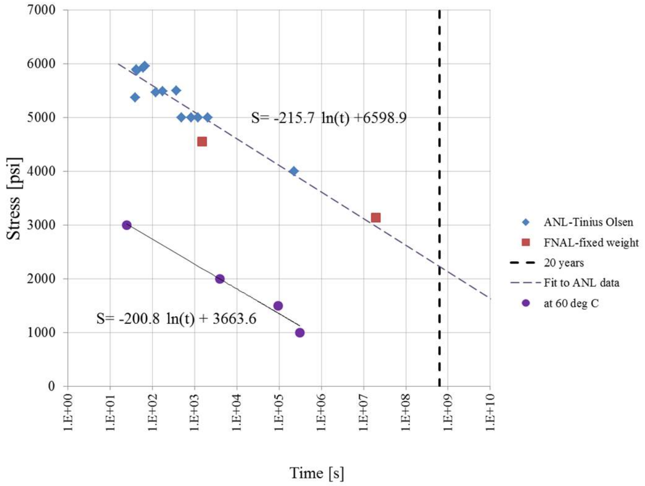


FIGURE 16. CREEP RUPTURE PLOT OF WEB SPECIMEN.

knitlines.
E. Brittle failure.
G. Brittle failure along the knit line leaving a very flat surface but with signs of stronger bonding.

From the data, the best knit lines are made at the highest melt temperature with the breaker plate and new screw having no significant effect.

Figure 13 shows the fracture surfaces of each failed knit from extrusion sample C plus the inclusion of the non-knit line ductile failure of web 3 for reference. Consistent with the discussion in section 3 and the classification of fracture surfaces in section 7, the fracture surfaces reveal less deformation and therefore reduced bonding strength in the interior webs . Likewise the outer most cells, 4 and 12 reveal significant deformation while at the same time exposing the knit line interface. This deformation is consistent with better bonding.

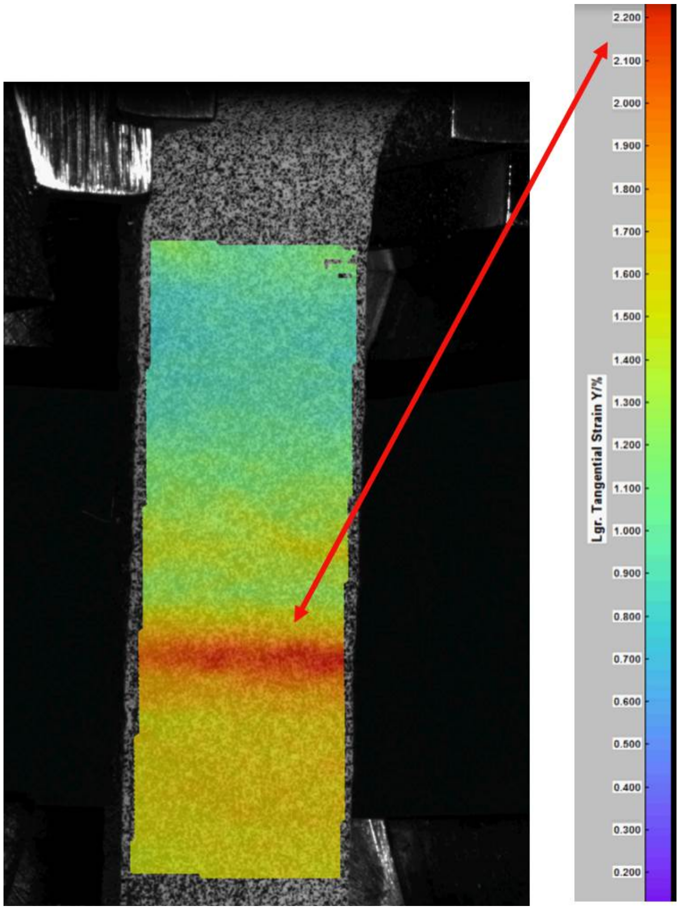


FIGURE 17. OPTICAL STRAIN MEASUREMENT OF A WEB 7 SAMPLE PRIOR TO FAILURE SHOWING THE STRAIN TO BE 2̃.2 % IN THE KNIT AREA.

CONCLUSION

A test was developed to both evaluate and characterize and potentially improve the knit line strength in a complex extrusion profile. The resulting web test is an easy and reliable quantifiable test of knit line strength for web middle knits.

Based on tests with varied conditions, it is concluded that the web knits are generally as strong as the base material with some exceptions. The stress reached is at the level of the yield stress even when large scale yielding is not present. Samples made at higher temperatures have improved ductility. In general the samples exhibit better ductility (necking and reasonable elongation) at the outer webs compared with very little to no ductility for the interior cells. The extent of the interior cells depends on the melt temperature. The samples generally fail at the knit line. Samples failing outside the knit line occur only in the outer cells and the total elongation in these cases is greater than when failing at the knit line.

The visual appearance of the interface reveals a qualitative state of the adhesion at the knitline. Very brittle knitline failures exhibit extremely smooth fracture surfaces. These surfaces exhibit zero ability to sustain inelastic deformation across the knit line. While very smooth failure surfaces appear to be unbounded and potential material voids, tests indicate that there is significant strength transmitted across the smoothest knit lines. In some cases, these knit lines fail at stress below the bulk material yield (5400psi versus 6000 psi) and exhibit negligible inelastic strain but there is no evidence supporting either complete lack of bonding or a material void. The latter is a great concern due to the potential for slow crack propagation over time. It is also difficult to conclude much about the knit line strength by examining the percent of smooth versus non-smooth area. Samples that exhibit good ductility across the knit line often exhibit distinct surface zones though these are generally less smooth than the brittle failures.

ACKNOWLEDGMENT

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