TP-H1148 KNITLINE INTEGRITY EVALUATION

FINAL REPORT

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1.0 BACKGROUND

Approximately 40 propellant mixes are to be cast to complete a segment. The casting is ordinarily a continuous operation, so that, after a mix has been cast, a subsequent mix is cast on top of it, forming a knitline between two propellant surfaces. The present acceptable casting interruption is only three hours (about 6 1/2 hours after ECA addition), and there is no clear definition as to the course of action should longer cast delays occur. The reason for this present three-hour time constraint is the concern for possible degradation of the knitline properties.

Previous studies have been conducted (mainly by Ned Caldwell, References 2 - 7) to evaluate the use of epoxy primers applied to the knitline to improve the knitline integrity after an extended delay is encountered. The conclusion that epoxy primer is essential to the knitline was based on 2-Inch per minute/77°F standard mechanical properties data. Also in those studies, propellant that was used came from 5-gallon mixes wherein the fresh, or "top" propellant was cast on top of the "bottom" propellant immediately after the end of mix of the 5-gallon mix cycle, which is about 1.5 hours after ECA mixing.

The original objective of ETP-0340, "TP-H1148 Knitline Integrity Evaluation" (Reference 8) was to reevaluate the TP-H1148 propellant knitting capabilities due to minor changes in TP-H1148 raw materials and to test the effects of aging on the knitline and the bondline of the propellant to the liner (when epoxy primer has been applied). Most of the tests in ETP-0340 had been designed to include the use of GE-100 epoxy primer, but during the testing it was found that primer application does not improve the strain endurance capability, increases the variation in the tensile data, and forms a hard propellant layer at the bondline. Thus it was decided to conduct most of the testing without the epoxy primer.

2.0 OBJECTIVE

To provide information necessary to determine what approach should be taken when an extended interruption in casting occurs.

3.0 SUMMARY

An extensive study of the knitline capability of TP-H1148 RSRM propellant was conducted. The study consisted of six parts, each one of them to test different parameters or to duplicate/verify the results using other propellant evaluations (sets of raw materials).

Since it was intended to closely simulate a production cast delay, for most of the testing propellant was used from 600-gal mixes, keeping the "bottom" propellant under vacuum and elevated temperature. Pains were taken to test the knitline mechanical properties at realistic "cooldown", or thermal loading, conditions; i.e., at low rates and by testing strain endurance properties.
The above production-oriented approach was one of the reasons to eliminate the GE-100 epoxy primer, which otherwise causes a 30-hour segment downtime and also creates a hard propellant layer somewhere in the segment.

4.0 CONCLUSIONS

1. The knitline between two adjacent propellant mixes can be detected starting at 8 hours from ECA addition (7 hours from ECA mixing) into the bottom propellant. After this time, differences in the mechanical properties become apparent.

2. Degradation of the 2 ipm/77°F strain at maximum stress ($\varepsilon_{m}^{2^6}$) below the specification criteria (30%) occurs only after 18 hours from ECA addition of the bottom propellant. Very long cast delays (more than 30 hours) cause a 40-50% reduction of the strain endurance capability and therefore cannot be tolerated in the forward segment transition zone and are very detrimental in other locations and in other segments. This means that there is not a good solution to longer cast delays.

3. There is no significant reduction of the maximum stress ($\sigma_{m}$) even up to 96 hours of cast delay. Strain at failure ($\varepsilon_{f}^{2^6}$ 2 ipm and 77°F) and strain endurance (2%/day) are much more sensitive to the knitline than the strain at maximum stress ($\varepsilon_{m}^{2^6}$). A low extension (crosshead) rate (0.002 ipm) has the same sensitivity to knitline as the standard 2 ipm extension rate.

4. When the knitline is first detectable (after 8 hours from ECA addition), the strain CV (coefficient of variation) out of six dogbones begins to increase drastically from about 3% for control samples of TP-H1148 to 10% for an 18 hour cast delay knitline.

5. There are some differences regarding the degradation of the knitline strain capability with cast delay time for different propellant evaluations (and/or different testing series). For example, Knitline VI strain capability degraded faster than in the other knitline studies.

6. The fresh (top) propellant ECA reaction time; i.e., from beginning of propellant mixing, should be above 3 hours at the time of casting in order to reduce the migration of ECA from the fresh propellant to the "bottom" propellant, which reduces the knitline capability. There is only a slight additional improvement when the reaction time of the fresh propellant is 4, 5, and 6 hours.
7. Application of GE-100 epoxy primer, 1.5 mil thick, creates a very hard and brittle 0.16-inch thick layer of propellant. Therefore, primer is not recommended. GE-100 washcoat does not improve the knifeline strain endurance capability and adds 30 hours (tooling breakdown and set up time) to the segment production time. There are also some inherent problems such as control of the primer thickness, contamination of the liner adjacent to the propellant interface with epoxy primer, and postcuring and aging reactions which are typical of epoxy-rich formulations.

5.0 RECOMMENDATIONS

1. For the transition zone of the forward segment (approximately mix numbers 17 - 23), only 8 hours of cast delay after ECA addition (7 hours after ECA mixing) is allowed.

2. For other locations in the forward segment and all regions of the aft and center segments, 18 hours of cast delay after ECA addition (17 hours after ECA mixing) is allowed.

3. If an interruption longer than 3 hours occurs, prepare a set of 1/2-gallon cartons with knifeline in order to help in selling the segment. That is, using production propellant, prepare half-filled cartons; hold at conditions simulating segment casting interruption as to time, vacuum, temperature; complete carton casting with fresh propellant.

4. When resuming the casting after longer than 3 hours interruption, the fresh propellant should not be cast into the motor before 2 hours after end of mixing (3 hours is preferred).

5. Do not use epoxy primer (GE-100) when longer cast delays occur. In such a case we should try to sell the segment on an individual basis by calculating the safety factor at the location of the knifeline, and we may find it necessary to certify the segment only for limited use, such as summer launch or static firing.

6. Top management should be aware that a long cast delay may result in scrapping a segment and therefore should be willing to take calculated risks and try to resume casting quickly, as we did during the M-24 mixer fire in 1985.
6.0 DISCUSSION

The knitline study consisted of six series of testing (Knitline I to Knitline VI). In each series a certain parameter was evaluated or the previous testing was just repeated with another material evaluation. Even though the ECA reaction begins only when propellant mixing begins, we stayed with the historical definition, i.e., cast delay time begins from ECA addition. The difference between ECA addition and ECA mixing is about an hour.

Knitline I - The knitline capability at 24, 30, 36, 48, 60, 72 and 96 hours cast delay was tested with and without GE-100 epoxy primer. The fresh (top) propellant age was two and four hours after its ECA addition. The propellant was prepared in 600-gal mixers (bottom) and M-209 5-gal mixer (top), and no vacuum was applied when the bottom propellant was cured in the oven.

The results of knitline I are summarized in References 9 and 10. The most important results of this study are presented in Figure 1. Strain endurance testing (2%/day) is more sensitive to cast delay time and epoxy primer application than the standard strain at maximum stress ($\varepsilon_{ma}$ at 2 ipm). The strain endurance test better simulates the thermal load caused by slow cooldown of a RSRM segment. The knitline with 96 hours cast delay with GE-100 epoxy primer has lower strain endurance capability (only 15% in comparison to 19%) than without the primer, even though the strain at maximum stress ($\varepsilon_{ma}$) is higher with the primer. Fresh (top) propellant at an age of four hours from ECA addition has better strain capability ($\varepsilon_{ma}$) than fresh propellant at an age of two hours, in particular for samples without primer. There is no significant reduction of maximum stress ($\sigma_m$) even at 96 hours cast delay.

In summary, there is a very significant reduction of the strain endurance capability from 31.5% for the control (no knitline) to 15-19% strain at 60-96 hours cast delay. Most of the reduction occurs during the first 24 hours of cast delay.

Knitline II - The knitline capability at 4, 6, 8, 10, 12, 14, 16, 18, 24, 28, 32, 36, 42 and 46 hours cast delay was tested without primer. The fresh (top) propellant was cast four hours after its ECA addition. To study cast delays up to 18 hours, 600-gal production propellant mixes were used. For longer cast delay times, the top propellant was prepared with a 5-gal mixer. No vacuum was applied when the bottom propellant was cured in the oven.

The results of Knitline II are summarized in Reference 11. The most important results are presented in Figures 2, 3, and 4.

Strain at failure ($\varepsilon_{fa}$) is much more sensitive to the knitline than the strain at maximum stress ($\varepsilon_{ma}$). The knitline can be detected after 8 hours when a deviation from the range of the control mixes begins (Figure 4). The deviation from the QC acceptance criteria, that is, a minimum of 30 percent strain ($\varepsilon_{ma}$) for TP-H1148 propellant, begins only after 26 hours of cast delay (Figure 3). The knitline stress capability (Figure 2) is as good as the control mixes (no knitline) up to 42 hours of cast delay.
Knitline III - The knitline capability at 4, 6, 8, 10, 12, 14, 16 and 18 hours cast delay was tested without GE-100 primer. The fresh (top) propellant age was four hours after its ECA addition. All propellants were 600-gal production mixes, and the bottom propellant was held under vacuum and elevated temperature in order to simulate real production conditions. The results are presented in Table 1 and Figures 5-10. Again the knitline can be detected after 8 hours after ECA addition when a deviation from the range of the control mixes begins (Figure 5 for \( \epsilon_m^{28} \) and Figure 6 for strain endurance).

The deviation from the QC acceptance criteria (\( \epsilon_m <30\% \)) begins only after 18 hours of cast delay (Figure 5). The knitline stress capability (Figure 7) is as good as, and even 3.5 psi higher than, the control mixes.

Testing at a very low extension rate (Figures 8 and 9) (0.002 lpm) gives knitline/cast delay characteristics similar to those at the standard 2 lpm extension rate, thus proving that the knitline has a good bond at the very low rate since any flaw has enough time to propagate. After 8-10 hours of cast delay when the knitline between the top and bottom propellants is first detectable, the coefficient of variation (CV) of the strain (out of six dogbones) begins to increase, from 2-3\% for the control mixes up to 10-15\% at 18 hours of cast delay (Figure 10). This drastic increase in the variation of the knitline strain capability is also important for safety factor calculations, and at least for the critical transition zone in the forward segment it is recommended to allow cast delays from ECA addition of no greater than 8 hours.

Knitline IV - The effects of the fresh (top) propellant age on the knitline capability were tested when the bottom propellant with about 10 hours cast delay was held under vacuum and elevated temperature. Again, 600-gal. productions mixes and no GE-100 primer were used. The results of Knitline IV are summarized in Reference 12 and Figures 11 - 15.

Fresh (top) propellant age is an important parameter influencing the knitline capability. Fresh propellant at an age of two hours has inferior mechanical properties in comparison to those at 3, 4, 5, and 6 hours age. The fact that 2-hour "fresh" propellant produced inferior knitline mechanical properties may be explained by diffusion of ECA from the "fresh" propellant into the bottom propellant, thus decreasing the amount of ECA present to react with HB polymer in the top, fresh propellant adjacent to the knitline, thereby reducing the knitline mechanical properties.

The above phenomenon might explain why previous studies by N. Caldwell (References 2 - 7) in which fresh (top) propellant at only 1-1/2 to 2 hours age had been used, recommended the use of GE-100 epoxy primer. Again it can be seen that the knitline mechanical properties are more sensitive to strain at failure (\( \epsilon_m^{28} \) and strain endurance [2\% strain/day]) than to minimum strain at maximum stress (\( \epsilon_m^{28} \)) and maximum stress (\( \sigma_m \)). Stress values were as usual even higher (by about 6 psi) than the propellant without the knitline, while the strain at failure and strain endurance were lower than the control mixes. The CV (coefficient of variation) out of six dogbones is much higher for the two hours fresh propellant age, and only after four hours age does the CV reach the control sample level (Figure 15).
The conclusion from this study is that TP-H1148 propellant should not be cast into the segment as soon as possible after mixing as was previously thought. In case of interruption in the casting operation, the 'fresh' propellant age should be a minimum of 3 hours old, and it would preferably be about 4 hours old. The fresh propellant age should be counted from the beginning of mixing of the ECA and not the addition time of ECA. This means that the recommended actual fresh propellant age should be about 3 hours, and in any case no less than 2 hours, from end of mixing.

Knitline V - The knitline capability at 8, 10, 12, 14, 16, and 18 hours cast delay was tested. Only at 10 and 16 hours was GE-100 primer applied. The fresh (top) propellant age was four hours after its ECA addition. All propellants were 600-gal. production mixes, and the bottom propellant was held under vacuum and elevated temperature to simulate real production conditions.

The purpose of Knitline V was to duplicate Knitline III tests with another propellant evaluation and to verify again the effect of GE-100 epoxy primer.

The results are summarized in Table 2 and plotted in Figures 16 -19. The deterioration of the knitline capability is higher for Knitline V (E68 evaluation) than for similar testing in Knitline III (E67 evaluation) with the same characteristics: the knitline now was detected after 6 (rather than 8) hours of cast delay as the rupture strain ($\varepsilon_{r}$) and the strain endurance (2%/day) begin to depart from the control samples. The strain at maximum stress ($\varepsilon_{m}$) is lower than the QC acceptance criteria of 30% after 16 (rather than 18) hours cast delay. The maximum stress ($\sigma_m$) of the knitline is as good as or better than the control samples. The strain CV calculated out of six dogbones increased from about 3% for the control sample to about 10% for the knitline at 18 hours cast delay. As before, the departure from the control sample range begins at 9 - 10 hours cast delay.

Application of GE-100 primer did not improve the knitline capability. At 10 hours cast delay the strain capability is even lower than the unprimed knitline, while at 16 hours cast delay there was some improvement due to the primer application. Note that the CVs of $\varepsilon_{r}$ are high for the dogbones with the primer, probably due to addition variable. The hardness of the epoxy-primed surfaces is much higher (85 Shore A) in comparison to about 60 Shore A for the control TP-H1148 propellant. This hard propellant layer (about 0.16 inch thick) is discussed in Knitline VI.

Knitline VI - The knitline capability at 6, 10, 16, 24, (48), 72, and 96 hours of cast delay was tested in two parts. The first part was similar to Knitlines III and V in which 600-gal. production propellant was held under vacuum and elevated temperature until 6, 10, and 16 hours cast delay without epoxy primer. In the second portion, a long cast delay was simulated in which the bottom propellant (a 600-gal. production mix) was held up to 24, 48, 72, and 96 hours total cast delay, fresh propellant (from 5-gal. mixes) was cast both with and without GE-100 primer. (Due to weighment error, the 48 hours mix was out of specification and scrapped.)
Knitline VI is summarized in Reference 13. The Knitline VI strain capability departed from the control samples faster than any of the previous knitline studies (Figures 20 - 26), and after 72 hours cast delay the strain endurance (2%/day) was only 17% in comparison to 31% for the control TP-H1148 propellant. The maximum stress ($\sigma_m$) of the knitline was as good as the control samples, even at 96 hours cast delay (Figure 24).

Although the use of primer did improve $\varepsilon_m^{2.8}$, $\varepsilon_m^{1}$, and $\varepsilon_1$ strains (2 ipm/77°F data), no improvement was observed in strain endurance (2%/day) which better represents the thermal loads which develop during segment cooldown. The strain CV (coefficient of variation out of six dogbones) for the epoxy-primed knitline is significantly larger than the unprimed knitline (Figure 25). Addition of primer created a hard propellant layer adjacent to the knitline (see penetrometer readings in Figure 26). A 0.16-in. thick, hard/brittle propellant layer within a segment is undesirable, and therefore the addition of primer is not recommended. Primer application is not a completely controlled operation (thickness variation will occur), and there are inherent problems of liner contamination with epoxy primer, and later on, with post-curing reactions of the excess epoxy at the liner bondline and the propellant knitline. An additional advantage of not using primer is that it would not involve any of the difficulties of removing casting tooling, applying primer, and reassembling tooling, all of which constitutes an extra 30 hours in downtime during segment production (Reference 11).

To estimate the mechanical properties of the hard propellant layer created by GE-100 primer, a correlation was established between the mechanical properties and the hardness of TP-H1148 propellant. In Table 3, there is a summary of the linear regression constants and correlation coefficients for the last nine raw material standardizations.

The average correlations for TP-H1148 propellant are:

$$
\sigma_m = -83.87 + 3.649 \text{ (Shore A)}, \ R^2 = 0.985
$$

$$
\varepsilon_m^{2.8} = 48.4 - 0.2405 \text{ (Shore A)}, \ R^2 = 0.918
$$

$$
\varepsilon_1^{2.8} = 91.47 - 0.7807 \text{ (Shore A)}, \ R^2 = 0.976
$$

$$
E^{2.8} = 695.2 + 23.11 \text{ (Shore A)}, \ R^2 = 0.987
$$
As can be seen from the above table, there are very good correlations between TP-H1148 propellant hardness (10-second Shore A) and the mechanical properties. In Table 4, there is a comparison between typical TP-H1148 mechanical properties and the estimated mechanical properties of the hard (Shore A = 85) propellant layer when GE-100 primer is used. The hard propellant layer has only half of the strain at failure, double the strength (stress), and much higher modulus (by a factor of 2:3). The hardness of the primed propellant knitline was measured for several dogbones, and there was a scattering of Shore A values between 75 and 92. This shows that even more extreme mechanical properties exist than those estimated at a Shore A hardness of 85. It should be noted that the above correlations may be used in the RSRM program to quickly estimate the mechanical properties of a segment by simple measurement of the 10-second Shore A hardness. (For example, this can be done before fin popping/removal operations, or to estimate the mechanical properties of the segment at any time without cutting out and testing tensile specimens.)
REFERENCES

1. MEMO EDB-72-85, "Knitting Tests with Poseldon TP-H1148 Propellant", R. D. Wallace
7. TWR-12578, "GE-100 Epoxy as a Replacement for EPON-812", N. A. Caldwell
8. ETP-0340, "TP-H1148 Knitline Integrity Evaluation", K. A. McCoy
10. Memo 2432-FY90-M028, "TP-H1148 Knitline Integrity Evaluation - GE-100 Primer Application Strain Endurance", K. A. McCoy
11. Memo 2432-FY90-M068, "TP-H1148 Knitline Integrity Evaluation - Cast Delay at Ambient Pressure", K. A. McCoy
12. Memo 2432-FY90-M067, "TP-H1148 Knitline Integrity Evaluation - 10-Hour Cast Delay", K. A. McCoy
Table 1. Data for Knitline Study III

<table>
<thead>
<tr>
<th>Sample</th>
<th>$E_{2.6}$ (psi)</th>
<th>$\sigma_m$ (psi)</th>
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Strain Endurance 2%/day

- $\sigma_0$ = 70.3
- $\varepsilon_0$ = 77 oF
- $\varepsilon^n$ = 0.002

x-head rate = 0.002 bpm

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REVISION ______________

FORM TC 7964-310 (REV 2-88)

TWR-18755
Table 2. Knitline V Test Results

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<td>17</td>
<td>12.6</td>
<td>11.9</td>
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1. STRAIN ENDURANCE (S.E.) 2%/day
2. EACH DATA POINT IS AN AVERAGE OF SIX DOGBONES TESTED AT 2IPM/77 DEG. F.
3. E680217 WAS THE BOTTOM PROPELLANT.
4. INDEX A REPRESENTS CONTROL 1/2 GALLON CARTON OF TOP PROPELLANT.
5. INDEX 1 AND 2 REPRESENT TWO DIFFERENT CARTONS (DUPLICATES) OF A KNITLINE.
6. INDEX 3 AND 4 REPRESENT TWO DIFFERENT CARTONS (DUPLICATES) OF A KNITLINE WITH 1 MIL GE-100 EPOXY PRIMER
7. 4-14-1 MEANS: 14 HOURS CAST DELAY WHEN THE FRESH (TOP) PROPELLANT WAS CAST 4 HOURS AFTER ECA ADDITION

*VOIDS CAUSED EARLY FAILURE
### Table 3. Linear Regression Analysis* of TP-H1148 - Mechanical Properties Versus Shore A

<table>
<thead>
<tr>
<th>EVAL. NO.</th>
<th>$C_m$ (psi)</th>
<th>$C_m^{2.6}$ (%)</th>
<th>$C_T^{2.6}$ (%)</th>
<th>$e^{2.6}$ (psi)</th>
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</thead>
<tbody>
<tr>
<td></td>
<td>A</td>
<td>B</td>
<td>R²</td>
<td>A</td>
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<tr>
<td>E66</td>
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<td>E68</td>
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<td>E69</td>
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<td>3.800</td>
<td>0.980</td>
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<td><strong>1CV(%)</strong></td>
<td>29.3</td>
<td>11.8</td>
<td>1.53</td>
<td>6.8</td>
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</table>

The values which are underlined were not taken in the calculation of the average.

*For example: $e^{2.6} = -695.2 + 23.11$ (Shore A), $R^2 = 0.987$
Table 4. Comparison Between Typical TP-H1148 Propellant Mechanical Properties and The Hard Propellant Layer When Epoxy Primer Is In Use

<table>
<thead>
<tr>
<th></th>
<th>$\sigma_m$ (PSI)</th>
<th>$\varepsilon_m^{2.5}$ (%)</th>
<th>$\varepsilon_f^{2.5}$ (%)</th>
<th>$E^{2.5}$ (PSI)</th>
</tr>
</thead>
<tbody>
<tr>
<td>TYPICAL TP-H1148 PROPELLANT</td>
<td>110</td>
<td>36</td>
<td>48</td>
<td>550</td>
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<tr>
<td>KNITLINE WITH EPOXY PRIMER</td>
<td>226</td>
<td>28</td>
<td>25</td>
<td>1269</td>
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</table>
Figure 1. SRM Knitline Integrity Evaluation - Strain Properties Summary
Figure 2. TP-H1148 Nthline Evaluation – Mechanical Properties Summary

- CAST DELAY (hours)
- Top casting with 4-hour "fresh" propellant
- Reference (Without Nthline)
- 135°F, break vacuum, & no GE-100 primer
Figure 4. TP-H148 Knitline Evaluation - Mechanical Properties Summary
Figure 5. Strains at 2.0 in./min With Cast Delay Time - Knitline Study III
Figure 7. Maximum Stress at 2.0 in./min With Cast Delay Time - Knitline Study III
Figure 8. Strains at 0.002 in./min With Cast Delay Time - Knitline Study III
Figure 9. Maximum Stress With Cast Delay Time - 0.002 in./min (Knitline Study III)
Figure 10. Coefficients of Variation For Strains With Cast Delay Time - Knitline Study III
Figure 11. SRM Knitline Integrity Evaluation - Second Method Mechanical Properties Summary
Figure 14. SRM Knitline Integrity Evaluation - Second Method Mechanical Properties Summary
Figure 15. Coefficients of Variation for C_m with Fresh Propellant Age - Knitline Study IV
Figure 16. Strains With Cast Delay Time - Knitline Study V
Figure 17. Strain Endurance With Cast Delay Time - Knitline Study V
Figure 18. Maximum Stress with Cast Delay Time – Knitline Study V
Figure 19. Coefficients of Variation For Strains With Cast Delay Time
Knitline Study V
Figure 22. TP-H148 Knitline VI Evaluation Mechanical Properties Summary
Figure 24. TP-H1148 Knitline VI Evaluation Mechanical Properties Summary
Figure 25. Coefficients of Variation For Strains With Cast Delay Time
Knitline Study VI

- knit line (no primer)  • knit line (with primer)  + control samples (no knit line)
Figure 26. TP-H148 Knitline VI Evaluation Mechanical Properties Summary