Executive summary

Validation of F-16 wing attachment fitting bolts

Example of surface rusting of WAF bolt ends and nuts

Problem area
During a modification programme, the Wing Attachment Fitting (WAF) cadmium-plated steel bolt assemblies from 33 F-16 aircraft were cleaned in a non-approved chemical solution, a paint stripper. This removed much of the cadmium plating, leading to in-service superficial rusting of some bolt ends and nuts. The OEM and chemical manufacturer became concerned about possible hydrogen embrittlement of the bolts owing to exposure to the paint stripper. The OEM recommended tensile and stress durability hydrogen embrittlement testing of some of the bolts before continuing to fly with the rest. In the meantime, all 33 aircraft were temporarily grounded.

Description of work
The Royal Netherlands Air Force (RNLAF) sent the bolts from 5 aircraft to the NLR, and 50 bolts were selected for tensile testing.

Results and conclusions
The tensile test data, statistical analysis, fractography, and a discussion of electrochemical and hydrogen diffusivity considerations and the “no failure in 200 hours” criterion, led to the conclusion that the bolt failure strengths were unaffected by the paint stripper. The aircraft were cleared for further operation, whereby all the remaining WAF bolt assemblies were cleaned in an approved cleaner, protected with primer, and replaced at the latest during the next scheduled inspection.
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Customer National Aerospace Laboratory NLR
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Distribution Unlimited
Classification of title Unclassified

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25/12/2011
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Validation of F-16 Wing Attachment Fitting Bolts

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During a modification programme, the Wing Attachment Fitting (WAF) cadmium-plated steel bolt assemblies from 33 F-16 aircraft were cleaned in a non-approved chemical solution, a paint stripper. This removed much of the cadmium plating, leading to in-service superficial rusting of some bolt ends and nuts. The OEM and chemical manufacturer became concerned about possible hydrogen embrittlement of the bolts owing to exposure to the paint stripper. The OEM recommended tensile and stress durability embrittlement testing of some of the bolts before continuing to fly with the rest. However, in the meantime all 33 aircraft were temporarily grounded. The Royal Netherlands Air Force (RNLAF) sent the bolts from 5 aircraft to the NLR, and 50 bolts were selected for tensile testing only. The results, statistical analysis, fractography, and a discussion of electrochemical and hydrogen diffusivity considerations and the “no failure in 200 hours” criterion, led to the conclusion that the bolt failure strengths were unaffected by the paint stripper. The aircraft were cleared for further operation, whereby all the remaining WAF bolt assemblies were cleaned, protected with primer, and replaced at the latest during the next scheduled inspection.

Keywords: H-11 steel, hydrogen embrittlement, tensile test, fractography

Introduction

Scheduled inspections at two Royal Netherlands Air Force (RNLAF) bases revealed surface rusting of bolt ends and nuts in the Wing Attachment Fitting (WAF) steel bolt assemblies of 3 F-16 aircraft[1], for example Fig. 1. Checks showed that all three aircraft had taken part in a wing modification programme, and that during this programme the WAF bolts, nuts and washers had been immersed for up to 3 days in a bath of paint stripper, which is a non-approved cleaning liquid. The paint stripper removed significant amounts of the cadmium plating originally applied to the bolts, nuts and washers.

Further checks showed that 33 aircraft had been through the wing modification programme such that the WAF bolt assemblies were most probably immersed in the paint stripper.[1] The OEM was informed, and in consultation with the manufacturer of the paint stripper expressed concern about possible hydrogen embrittlement of the bolts owing to the action of the paint stripper. The OEM recommended tensile testing and stress durability embrittlement testing (200 hours proof testing for internal hydrogen embrittlement, IHE) of some of the bolts before continuing to fly with the rest.

An RNLAF Crisis Management Team (CMT) then grounded all 33 aircraft.[1] The NLR was requested to advise on the OEM’s proposal and conduct a validation test programme on the suspect bolts, with the objective of allowing the CMT to clear the aircraft for operation. The NLR and members of the CMT agreed to a programme of tensile testing only. Proof testing was considered unnecessary, for reasons given later in this paper.
The validation programme comprised the following steps:

- Tensile tests and fractography
- Statistical analysis of the tensile test data
- Electrochemistry and hydrogen diffusivity considerations
- The proof test “no failure in 200 hours” criterion.

### Tensile test programme

The NLR received 160 suspect WAF bolt assemblies from 5 aircraft (32 sets per aircraft). Table 1 gives information on the bolt assemblies, which were selected to provide a wide range in flight and service hours since the paint stripper treatment. Each aircraft makes between 150 – 200 flight hours per year, with an average of over 40 hours between flights, so that the minimum service hours would have been more than 200.

#### Table 1  Aircraft and Suspect Bolt Assembly Information

<table>
<thead>
<tr>
<th>Aircraft</th>
<th>Flight hours since paint stripper treatment</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>536.1</td>
</tr>
<tr>
<td>B</td>
<td>441.4</td>
</tr>
<tr>
<td>C</td>
<td>87.8</td>
</tr>
<tr>
<td>D</td>
<td>27.9</td>
</tr>
<tr>
<td>E</td>
<td>6.1</td>
</tr>
</tbody>
</table>

In addition, 4 new bolts and 4 non-suspect (cadmium-plated) used bolts were supplied for a pilot test programme.

The bolt allocation per aircraft wing is as follows (D = bolt diameter):

- Upper WAFs: 6 × D12 (19.05 mm), 2 × D10 (15.88 mm)
- Lower WAFs: 6 × D14 (22.23 mm), 2 × D12 (19.05 mm).

The bolts were made from H-11 martensitic CrMoV steel per AMS 6487, heat-treated to a minimum tensile strength of 220 ksi (1517 MPa).

Custom-made clamping fixtures were prepared from 17-4 PH (H900) steel to enable tensile testing of complete bolt assemblies. The tests were done in an AVERY 1 MN static machine.

#### Pilot programme

Table 2 gives the results, which included one suspect bolt assembly. Note that the bolt from this assembly failed at a higher load than the new and non-suspect D10 bolts.

#### Table 2  Pilot Test Programme Results

<table>
<thead>
<tr>
<th>Aircraft</th>
<th>Bolt condition and type</th>
<th>Failure load (kN) and mode*</th>
</tr>
</thead>
<tbody>
<tr>
<td>–</td>
<td>new D14</td>
<td>547: ts</td>
</tr>
<tr>
<td>–</td>
<td>new D14</td>
<td>544: ts</td>
</tr>
<tr>
<td>–</td>
<td>new D12</td>
<td>413: ts</td>
</tr>
<tr>
<td>–</td>
<td>new D10</td>
<td>295: ts</td>
</tr>
<tr>
<td>unknown</td>
<td>used D14</td>
<td>583: ts</td>
</tr>
<tr>
<td>unknown</td>
<td>used D14</td>
<td>578: ts</td>
</tr>
<tr>
<td>unknown</td>
<td>used D12</td>
<td>425: b</td>
</tr>
<tr>
<td>unknown</td>
<td>used D10</td>
<td>307: b</td>
</tr>
<tr>
<td><strong>B</strong></td>
<td>suspect D10</td>
<td>312: b</td>
</tr>
</tbody>
</table>

* ts = thread-stripped; b = bolt-break
The three bolt-breaks, which occurred in the threaded parts of the shanks, were examined fractographically. The fractures were due to overload by a mixture of intergranular and transgranular dimpled rupture, for example the Scanning Electron Microscope (SEM) fractograph in Fig. 2. This type of overload fracture has been observed for other high strength steels.[2]

**Definitive programme**

For a proper statistical analysis it was required to test at least 30 bolts, preferably 50, having the same diameter. In view of this requirement and the bolt allocations per aircraft wing, the NLR selected 10 × D12 bolt assemblies from each of the 5 aircraft listed in Table 1. The selection ensured that the bolt assemblies came from upper and lower WAFs, and from right and left wings. Furthermore, the selection took account of any visible rust, i.e. bolt assemblies showing service-induced rusting were preferentially selected. In fact, only bolts from aircraft A and E showed any rust, which was light and only at the bolt ends. Note from Table 1 that these bolts had the longest and shortest flight hours, respectively, since paint stripper treatment. Thus rusting was not necessarily a progressive in-service phenomenon.

Figure 3 summarises the test data in a histogram. This gives the ranges in failure loads for the two failure modes, bolt-break (19 cases) and thread-stripped (31 cases). For each of the 5 aircraft the bolt-break failure mode resulted in minimum and average failure loads at least as high as those for the thread-stripped failure mode. Also, there is no apparent relation between the failure loads and flight hours since paint stripper treatment.

All the bolt-break failures were examined fractographically. The fractures were due to overload by a mixture of intergranular and transgranular dimpled rupture, as already illustrated in Fig. 2. There was no evidence of intergranular fracture owing to IHE (in particular, no evidence of IHE close to the thread roots).

**Statistical analysis of the tensile test data**

*Failure modes*

Statistical tests were done to determine whether the bolt-break and thread-stripped data sets could be pooled. The tests determined whether the differences in the means and variances of the data sets were statistically significant, with the level of significance set to a standard value of 0.01. A small-sample T-test on the means and a small-sample F-test on the variances showed that the differences in the means and variances were significant. Hence the data sets could not be pooled. Normal fits were made to the two data sets. Figures 4 and 5 show that a normal distribution fits both data sets. The goodness-of-fit was supported by the Kolmogorov-Smirnov and Hollander-Proschan test statistics.[3]

In combining data from all 5 aircraft, it can be seen from Figs. 4 and 5 that the bolt-break minimum and mean failure loads were well above those for the thread-stripped failures. The mean values were 437 kN and 422 kN, respectively.

*Flight hours since paint stripper treatment*  

The major differences in flight hours since paint stripper treatment (A and B versus C, D and E) are indicated by filled and open symbols in Figs. 4 and 5. There is no clustering of the failure loads according to flight hours, in agreement with a lack of a trend in Fig. 3.
Minimum strength level

Figure 6 depicts the same information for the bolt-break failure data as Fig. 4, but now in terms of a Cumulative Distribution Function (CDF), together with the failure strength A- and B-values. These represent the 95% confidence lower limits on the first and tenth percentiles of the property distributions, 399 kN and 415 kN, respectively. The confidence limits were determined using the Fisher information matrix.[4]

Figure 6 also shows the OEM failure strength A-allowable for D12 bolts, 395 kN. The estimated A-value from the bolt-break tests is higher, even though based on a limited data set of 19 cases. One may expect the A-value for a larger data set to be still higher.

Comparison of pilot and definitive programme data

The failure loads for the new and non-suspect used D12 bolts in Table 2 were 413 kN (thread-stripped) and 425 kN (bolt-break) respectively. These values are lower than the respective mean values, 422 kN and 437 kN, for the definitive programme data.

Summary

The results of the statistical analysis strongly suggest that the suspect D12 bolt assemblies have not undergone tensile failure load degradation owing to IHE caused by the paint stripper treatment. Furthermore, the fact that the bolt-break minimum and average failure loads were well above those for the thread-stripped failures suggests that the bolt shank material below the thread roots was undamaged. This is because (possible) hydrogen embrittlement of service-loaded bolts would be expected to damage the material subjected to stress concentrations below the thread roots, but not the threads themselves.

Electrochemistry and hydrogen diffusivity considerations

Figure 7 shows a simplified potential (E) – pH diagram for iron in water.[5] The two diagonal lines (a) and (b) bound the region of stability of water as a function of potential and pH. Above line (a) water is thermodynamically unstable with respect to the generation of oxygen gas. Between lines (a) and (b) water is thermodynamically stable. Below line (b) water is thermodynamically unstable with respect to the generation of hydrogen gas. Bubbles of hydrogen will evolve on a metal surface (acting as an electrode) in contact with water in this region.

The pH of the WAF bolt paint stripper is shown as a dashed vertical line in Fig. 7. Knowing that the bolts would initially be cadmium plated, and that cadmium plating is sacrificial with respect to steel, this E – pH diagram suggests that:

1. Depending on the cadmium-steel galvanic coupling potential, i.e. if it would be below about -0.6 V-SHE, hydrogen gas could be liberated at the cadmium/steel interface during the anodic attack and removal of the cadmium layer by the paint stripper. Some hydrogen could also be absorbed by the bolts, as discussed below.

2. The bolts would not corrode in the paint stripper, since its pH line is well outside the corrosion region. Note that the suggestion about no corrosion of the bolts is consistent with measurements of the open-circuit potential of another low alloy steel (4340) in paint strippers having pH values ranging from 8 – 11.5.[6] These measurements showed that the bare steel was in the water-stable (passivity) region of the E – pH diagram. In other words, even if all the cadmium plating on the WAF bolts were to be removed by
anodic attack, the bolts would remain uncorroded. (In fact, as mentioned earlier, inspection of all the suspect bolts before tensile testing showed no evidence of corrosion except service-induced light rusting on some of the bolt ends from the aircraft A and E.)

With respect to (1) above, there is the question of hydrogen absorption into the WAF bolts during the anodic attack and removal of the cadmium layer by the paint stripper. This could occur only if the galvanic coupling potential was below line (b), as may have been the case. For example, Fig. 8 shows the galvanic coupling potentials for cadmium and 4340 steel in several paint strippers, whereby it is seen that it is possible to transgress below line (b).

The only driving force for hydrogen absorption would be electrochemical, since the WAF bolts were not under mechanical stress during paint stripper treatment and the subsequent time before reinstallation in the aircraft structure. This means that much or all of the hydrogen that might have been absorbed could have diffused out through the bare steel surface once the bolts were removed from the paint stripper. An estimate of the time taken for any absorbed hydrogen to diffuse out of the bolts can be made as follows.

The checks mentioned in the introduction to this paper showed that the maximum time in paint stripper would have been no more than 3 days. The diffusion distance into the steel is given by \( X = \sqrt{D t} \), where \( D \) is the diffusion coefficient for hydrogen build-up in the steel and \( t \) is the time. The diffusion coefficients for build-up and decay of hydrogen in high strength steels are \( 2.2 \times 10^{-7} \) cm\(^2\)/s and \( 0.85 \times 10^{-7} \) cm\(^2\)/s, respectively.[7] Let \( X \) be the same distance during hydrogen build-up and decay. Then the time required for all the introduced hydrogen to diffuse out of the steel is given by:

\[
t_2 = t_1 \times \frac{D_1}{D_2}
\]

Substituting 72 hours for \( t_1 \) and \( 2.2 \times 10^{-7} \) cm\(^2\)/s and \( 0.85 \times 10^{-7} \) cm\(^2\)/s for \( D_1 \) and \( D_2 \), we obtain \( t_2 = 186 \) hours, which is slightly less than 8 days. This is less than the time between paint stripping and reinstallation in the aircraft structure, which the RNLAF stated to be typically 3 – 4 weeks.

Summarising, it appears unlikely that exposure of the WAF bolts paint stripper would lead to retention of any absorbed hydrogen by the time they were reinstalled in the aircraft. Nor were the bolts damaged by corrosion in the paint stripper, see point (2) above.

The proof test “no failure in 200 hours” criterion

As mentioned in the introduction to this paper, the OEM recommended stress durability testing (IHE proof testing) of some bolts. The OEM first suggested testing per ASTM Standard F519[8] and later per NASM1312-5[9], whereby suspect and new WAF bolts would be subjected to sustained tensile loads at 75 % of the notched fracture stress for 200 hours. Survival for 200 hours would validate the suspect bolts. This is the “no failure in 200 hours” criterion obtained from Troiano’s classic data.[10]

Figure 9 shows Troiano’s results for delayed failure of sharp-notched specimens of high strength 4340 steel after cadmium plating and baking for various times. There is obviously a strong effect of baking time on the delayed failure stress level. However, the most important point in the context of the present work is that most embrittled specimens failed well within 200 hours, in fact within 10 hours. This result is relevant to the service experience of the WAF bolts after paint stripper treatment. All these bolts had survived more than 200 service hours in the fully-torqued condition, making it very unlikely that they had been embrittled. This expectation, together with
the tensile test results and the electrochemical and hydrogen diffusivity considerations, led to the decision not to do stress durability testing. Subsequently it was possible to provide more justification for the omission of stress durability testing, see the postscript below.

Concluding remarks and remedial actions

The tensile test results and analysis, fractography, and a discussion of electrochemical and hydrogen diffusivity considerations and the proof test “no failure in 200 hours” criterion, led to the NLR’s conclusion that the WAF bolt tensile failure strengths were validated as unaffected by the paint stripper.

The CMT cleared the aircraft for further operation, whereby all the remaining WAF bolt assemblies were cleaned with an approved cleaner and protected with primer. These assemblies were subsequently replaced at the latest during the next scheduled inspection.

Postscript

Information obtained later from the OEM showed that the fully-torqued condition of the suspect bolts corresponded to 60 % of the OEM A-value allowable. In turn, this corresponds to 54 % of the mean failure load of the D12 bolt breaks shown in figures 4 and 6. At this 54 % level, equivalent to 160 ksi in figure 9, Troiano’s data cannot completely exclude failure of the bolts at service times beyond 200 hours, since his tests were stopped after 100 hours. However, additional data from tests up to 1000 hours \cite{6,11}, particularly the data from Movich \cite{11}, suggest that applying more than 50 % of the unembrittled failure load would have caused embrittled bolts to fail well within 100 hours.

Acknowledgements

CMT and NLR staff agreed to the validation programme on July 6, 2007. The 33 grounded aircraft were cleared for further operation on 13 July, 2007. This was made possible by the RNLAF’s prompt delivery of suspect bolt assemblies to the NLR; the ad hoc NLR testing team, Hotze Jongstra, Martin Martens, Marcel van der Kroeg, Niels Wildeboer, René Vermeulen and Ton van der Belt; and the CMT, particularly John Bronder, Chris Groot, and Stephan Verhoeven. The advice of Stan Lynch (DSTO Melbourne) is much appreciated.

References


Fig. 1  Example of a surface-rusted WAF bolt

Fig. 2  Fracture characteristics of the non-suspect used D12 bolt from the pilot programme
Fig. 3  Histogram of the tensile test data from the definitive programme: FH = Flight Hours since paint stripper treatment

Fig. 4  Normal fit for the bolt-break failure load data from the definitive programme
Fig. 5  Normal fit for the thread-stripped failure load data from the definitive programme

Fig. 6  Cumulative Distribution Function for the bolt-break failure load data from the definitive programme
Fig. 7  Simplified E – pH diagram for the iron-water system at 25 °C. E is the electrode potential with respect to the Standard Hydrogen Electrode (SHE)
Fig. 8  $E - pH$ diagram for (1) open circuit potentials of 4340 steel and (2) galvanic couple potentials for cadmium and 4340 steel in paint strippers [6].

$E$ is the electrode potential with respect to the Saturated Calomel Electrode (SCE):

$$E \text{ (SCE)} = 0.241 \times E \text{ (SHE)} \text{ at } 25 \degree C.$$  

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Fig. 9 - Delayed failure curves for various hydrogen concentrations corresponding to different baking times at 300 F (149 °C). Sharp notch specimens were used at the 230 ksi strength level [10].

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