

Metallurgical & Corrosion Testing, Failure Analysis and Consulting

Mr. Min Zhu (4)
Diamond Offshore Drilling, Inc.
15415 Katy Freeway, Suite 100
Houston, Texas 77094

222 Cavalcade Street
Houston TX 77009

P.O. Box 8768
Houston TX 77249

Telephone : (713) 526-6351
Direct : (713) 696-6222
Telefax : (713) 526-2964
E-mail : dick.jones@us.stork.com
Website : www.storksmt.com

Report No. 0091-10-19492R
Examination of Three Failed Inconel 718 Studs

Richard L. Jones
Senior Consultant

W. M. Buehler
Consulting Manager

March 1, 2010

INTRODUCTION

Stork Testing & Metallurgical Consulting, Inc. (ST&MC) received parts of three 3-inch diameter x 8 threads per inch Inconel 718 studs that had failed. The studs had been used in the construction of a Blow Out Preventer (BOP) stack built by the Shaffer Division of National Oilwell Varco (NOV) for Diamond Offshore Drilling, Inc. (DODI) in about 1999 and was used on DODI's *Ocean Confidence* drilling rig. The BOP stack consisted of three double BOPs each described as a Shaffer 18-3/4-inch 15M SLX. There were 20 identical studs used to connect the upper and middle double ram BOPs. Eleven of the studs had broken and the other nine had stripped the female threads from the top flange of the middle BOP. It was reported that the pre-load on each stud when the BOP was assembled was 605,089 lbf.

Also received was Shaffer Engineering Specification A-X010181 Revision A, Material Spec, High Strength Bolting UNS N07718 with Minimum YS of 150 Ksi and Maximum Hardness of 43 RC, dated January 15, 1999. The failed studs were reportedly manufactured to this specification and a new, unused stud was provided for comparison.

When the BOP was built twenty 16-inch long, 3-inch diameter Inconel 718 studs were used to connect the lower BOP to the middle BOP and also the middle BOP to the upper BOP. The top flanges of the lower and middle BOPs had 20 blind threaded holes. The bottom flanges of the middle and upper BOPs had 20 clearance holes. The studs were threaded into the top flanges and inserted through the bottom flanges with nuts applying the pre-load when tightened against the top surface of the bottom flanges.

On January 4, 2010, at around 5:30 PM, pressure testing to 15,000 psi was begun on the upper shear rams of the BOP in the test bay of the drilling rig. When the test pressure reached approximately 13,700 psi, all 20 studs at the connection between the upper and middle double ram BOPs suddenly failed. Eleven studs were found to be fractured and the remaining 9 studs pulled out of the top flange of the middle BOP, stripping the female threads.

Figure 1 is a view of fractured studs shortly after the failure. Figure 2 is a view of studs with stripped threads shortly after the failure. Figure 3 shows a view of the nuts on the ends of the studs on the top side of the bottom flange of the upper double BOP, before they were cut to remove the failed studs. Figure 4 shows the

upper surface of the top flange of the middle BOP, after disassembly of the BOP and before the pieces of the failed studs had been removed. Figure 5 shows the nine studs that pulled out of the threaded holes in the top flange of the middle BOP, with the stripped threads still attached.

The failed studs were numbered 1 through 20 by DODI personnel on the drilling rig. Studs 1 through 9 had stripped the female threads from the top flange of the middle BOP. Studs 10 through 20 had fractured. At a meeting with DODI, NOV and ST&MC on January 19, 2010, it was agreed that Studs 14, 15 and 16 would be examined by ST&MC initially.

In March 2004, as a result of problems others have had with Inconel 718 in sub-sea applications, the API adopted Specification 6A718, Specification of Nickel Base Alloy 718 (UNS N07718) for Oil and Gas Drilling and Production Equipment. This specification will be used as a reference in this report.

ST&MC was asked to determine the cause of the failure of the studs.

FINDINGS AND CONCLUSIONS

1. The Inconel 718 in Stud 14 was found to have grain boundary precipitates and intragranular acicular δ -phase in the microstructure. The presence of the acicular δ -phase made the alloy susceptible to hydrogen stress cracking (HSC). The hydrogen was likely a normal by-product of cathodic protection when the BOP was in service.
2. The reported pre-load equated to a tensile stress of 91,611 psi, which was 61 percent of the specified minimum yield strength (SMYS) required by Shaffer Engineering Specification A-X010181 for the studs and 56.6 percent of the measured yield strength of Stud 14.
3. The tensile properties, hardness and Charpy impact values of Stud 14 met the requirements of Shaffer Engineering Specification A-X010181 Revision A. However, the hardness of the unused stud and of Studs 15 and 16 was higher than specified.

4. The chemical composition of Stud 14 satisfied the requirements of Shaffer Engineering Specification A-X010181 Revision A, except that the niobium + tantalum content exceeded the maximum allowed. The niobium + tantalum content and the magnesium content exceeded the maximum allowed by API Specification 6A718. We do not believe these contributed to the failure.
5. The broken studs failed due to HSC of the Inconel 718 alloy while they were pre-loaded to approximately 61 percent of their SMYS. The embrittlement proceeded each time the BOP was in service and subjected to cathodic protection.
6. No evidence of fatigue cracking was found on the three fractured studs examined.

LABORATORY EXAMINATION

Visual Examination

Figure 6 is a view of failed Studs 14, 15 and 16, as received, along with the new stud and the nuts that had to be cut in order to remove the longer stud sections from the bottom flange of the upper BOP.

Figures 7 and 8 are views of the fracture surfaces on Stud 14. The red arrows indicate the location of the cracked threads shown later in Figure 11. Figures 9 and 10 are views of the fracture surfaces on Studs 15 and 16, respectively. The white arrows in Figures 7, 9 and 10 indicate the locations of the apparent fracture origins. Figure 11 is a view of cracked threads at the location indicated in Figures 7 and 8. The arrow indicates the location where a metallographic specimen was subsequently taken.

Even though the fractures were relatively flat and perpendicular to the longitudinal axis of the studs, which are characteristics of fatigue crack surfaces, none of the fracture surfaces on the three failed studs exhibited any of the other features typical of fatigue crack surfaces, such as ratchet marks around the edges or concentric progression marks across the surface.

The threads on the short sections of the failed studs were checked with a thread gauge just beyond the fracture surfaces. No evidence of stretching was found, except at the location shown in Figure 11.

Metallographic Examination

A metallographic specimen was taken across the cracked threads shown in Figure 11, at the location indicated by the arrow. Figure 12 is a photomicrograph of the section of the cracked threads, with arrows indicating the cracked thread and the fracture surface, shown in Figures 7 and 8. Figure 13 is a photomicrograph showing the cracked threads at a higher magnification. Arrows indicate the locations of smaller cracks.

Figure 14 is a photomicrograph showing the tip of the upper right hand crack shown in Figure 13. The crack is intergranular with acicular δ -phase present along grain boundaries and intragranular. This morphology is characteristic of cracks in some Inconel 718 after HSC.

Figure 15 is a photomicrograph of another section of the fracture showing a small longitudinal crack. Figure 16 shows the tip of the small crack, which is intergranular with acicular δ -phase present along grain boundaries and intragranular. Again, these features are typical of HSC in Inconel 718.

Figure 17 is a photomicrograph showing representative microstructure of Stud 14 with acicular δ -phase along the grain boundaries and through grains.

TENSION TESTING

A specimen from Stud 14 was prepared and tested in accordance with ASTM A 370. The results are shown below with the requirements of Shaffer Engineering Specification A-X010181 Revision A, and API Specification 6A718 for reference:

	<u>Stud 14</u>	<u>Spec. A-X010181</u>	<u>API 6A718</u>
Yield Strength*, psi	162,000	150,000 min.	120,000 min. 145,000 max.
Tensile Strength, psi	198,000	180,000 min.	150,000 min.
Elongation, % in 2 inches	19	12 min.	20 min. in 4D
Reduction of Area, %	36	15 min.	35 min.

*At 0.2% offset

The tensile properties of Stud 14 satisfied the requirements of Shaffer Engineering Specification A-X010181 Revision A, however, they did not meet the subsequent requirements of API Specification 6A718.

CHARPY IMPACT TESTING

Two sets of three Charpy specimens were prepared from Stud 14 and tested in accordance with API Specification 6A718. One set was tested at -26°F (-32°C) in accordance with Shaffer Engineering Specification A-X010181 Revision A, and one set was tested at -75°F (-60°C) in accordance with API Specification 6A718. The results are shown below:

<u>Specimen</u>	<u>Impact Value,</u> <u>ft-lbs at -26°F</u>	<u>Spec. A-X010181</u>	<u>Specimen</u>	<u>Impact Value,</u> <u>ft-lbs at -75°F</u>	<u>API 6A718</u>
1	28	13.3 min.	4	21	30 min.
2	28	13.3 min.	5	22	30 min.
3	21	13.3 min.	6	21	30 min.
Average	26	20 min.	-	21	35 min.

The Charpy impact values of Stud 14 satisfied the requirements of Shaffer Engineering Specification A-X010181 Revision A, however, they did not meet the subsequent requirements of API Specification 6A718.

HARDNESS TESTING

The three fractured studs and the new stud were tested for hardness using a Rockwell C indenter (HRC). The results are shown below with the requirements of Shaffer Engineering Specification A-X010181 Revision A, and API Specification 6A718 for reference:

	<u>HRC Values</u>	<u>Spec. A-X010181</u>	<u>API 6A718</u>
Stud 14	42, 41, 41	43 max.	32 min., 40 max.
Stud 15	44, 46, 47	43 max.	32 min., 40 max.
Stud 16	46, 44, 44	43 max.	32 min., 40 max.
New stud	44, 44, 44	43 max.	32 min., 40 max.

The hardness of the new stud and Studs 15 and 16 exceeded the maximum allowed by Shaffer Engineering Specification A-X010181 Revision A. The hardness of Stud 14 met the requirements of the specification, but none of the studs met the subsequent requirements of API Specification 6A718.

CHEMICAL ANALYSIS

The chemical analysis of Stud 14 was determined by optical emission spectroscopy, except for the carbon content, which was determined by combustion infrared absorption analysis. The results are shown below in mass percent along with the requirements of Shaffer Engineering Specification A-X010181 Revision A, and API Specification 6A718 for reference:

<u>Element</u>	<u>Stud 14</u>	<u>A-X010181</u>	<u>API 6A718</u>
Nickel, %	53.0	NS	50.0 – 55.0 max.
Cobalt, %	0.066	1.0 max.	1.00 max.
Nickel + Cobalt	53.1	50.0 – 55.0 max.	NS
Chromium, %	18.1	17.0 – 21.0 max.	17.0 – 21.0 max.
Molybdenum, %	2.86	2.80 – 3.30 max.	2.80 – 3.30 max.
Titanium, %	1.0	0.65 – 1.15 max.	0.80 – 1.15 max.
Aluminum, %	0.50	0.20 – 0.80 max.	0.40 – 0.60 max.
Carbon, %	0.020	0.08 max.	0.045 max.
Manganese, %	0.05	0.35 max.	0.35 max.
Silicon, %	0.11	0.35 max.	0.35 max.
Phosphorous, %	0.007	0.015 max.	0.010 max.
Sulfur, %	0.002	0.015 max.	0.010 max.
Boron, %	NT	0.006 max.	0.0060 max.
Copper, %	0.02	0.30 max.	0.23 max.
Lead, %	NT	NS	0.0010 max.
Selenium, %	NT	NS	0.0005 max.
Bismuth, %	NT	NS	0.00005 max.
Calcium, %	NT	NS	0.0030 max.
Magnesium, %	0.02	NS	0.0060 max.
Niobium + Tantalum, %	5.92	4.75 – 5.50 max.	4.87 – 5.20 max.
Iron, %	18.15	Balance	Balance

NT=Not Tested

NS=Not Specified

The chemical composition of Stud 14 satisfied the requirements of Specification A-X010181 Revision A, except that the niobium + tantalum content exceeded the maximum allowed. The niobium + tantalum content and the magnesium content exceeded the maximum allowed by API Specification 6A718.

DISCUSSION

The fractured studs failed due to HSC of the Inconel 718 alloy that was under stress at 61 percent of the SMYS and had a microstructure susceptible to hydrogen cracking due to the presence of acicular δ -phase in its microstructure. The results from the examination of Stud 14 made it unnecessary to examine Stud 15 and 16.

Inconel 718 is a precipitation-hardenable nickel-chromium alloy containing significant amounts of iron, niobium and molybdenum with lesser amounts of aluminum and titanium. It combines corrosion resistance and high strength with outstanding weldability. It is used in gas turbines, rocket motors, spacecraft, nuclear reactors and pumps. It has also been used offshore in the construction of sub-sea oil and gas equipment, however, it has been found that in order for it to be used successfully offshore it must be heat treated using a process that brings the niobium into solution and eliminates or minimizes grain boundary precipitates and intragranular acicular δ -phase in the microstructure. This is due to the fact that acicular δ -phase in the microstructure of Inconel 718 makes it susceptible to HSC when it is under stress. The source of hydrogen is the cathodic protection system used to protect most sub-sea oil and gas installations.

The δ -phase is a secondary phase in nickel base alloys. It can be globular or acicular, has an orthorhombic crystal structure and has a chemical composition described as Ni_3Nb type. The acicular δ -phase concentrates along the grain boundaries of nickel base alloys and provides a location where atomic hydrogen can accumulate and initiate brittle failure.

The key to eliminating or minimizing acicular δ -phase in Inconel 718 is its heat treatment. The table below compares the heat treatment specified in Shaffer Engineering Specification A-X010181 Revision A with that required by API Specification 6A718:

Engineering Specification A-X010181

1. Solution anneal at 1700 –1850°F and hold 1 to 2 hours at temperature.
2. Fast cool or water quench.
3. Age harden at 1325 – 1400°F for 8 – 10 hours and furnace cool to 1150 – 1200°F.
4. Hold for a total aging time of approximately 18 hours.

API Specification 6A718

1. Solution anneal at 1870 – 1925°F for 1 hour to 2-1/2 hours maximum.
2. Cool in air, water, polymer or oil.
3. Age harden at 1425 – 1475°F for 6 – 8 hours.
4. Air cool or faster to ambient temperature.

The higher solution annealing temperature required by API Specification 6A718 brings the niobium into solution and eliminates or minimizes grain boundary precipitates and intragranular acicular δ -phase in the microstructure.

Four factors are required for failure due to HSC to take place in Inconel 718:

1. A source of atomic hydrogen.
2. The presence of an applied load (such as the pre-load on a threaded fastener).
3. A high strength material (or high hardness as a reflection of strength).
4. A microstructure susceptible to HSC.

These four factors were present each time the BOP subject of this investigation was in sub-sea service. The source of atomic hydrogen was the cathodic protection system, which impressed a current on the BOP.

The applied load was the pre-load applied to the studs during the original assembly of the BOP. This load was 605,089 lbf on each stud.

The strength of the studs, as indicated by the testing of Stud 14 was approximately 162,000 psi yield strength, 198,000 psi tensile strength. The acicular δ -phase present in the microstructure made it susceptible to HSC under high stress.

Earlier failures of Inconel 718 used in sub-sea oil and gas equipment by energy companies prompted the need for a specification that would guide users to avoid the hydrogen embrittlement problem encountered in the present investigation. The API filled this need with Specification 6A718, Specification of Nickel Base Alloy 718 (UNS N07718) for Oil and Gas Drilling and Production Equipment, which was published in March 2004. Unfortunately, this was well after the failed studs were made.

We believe this completes the work that you requested. Please contact us if you have any questions or if we may be of further service.

Sincerely,



Richard L. Jones
Senior Consultant



W. M. Buehler
Consulting Manager

RLJ/WMB:kw

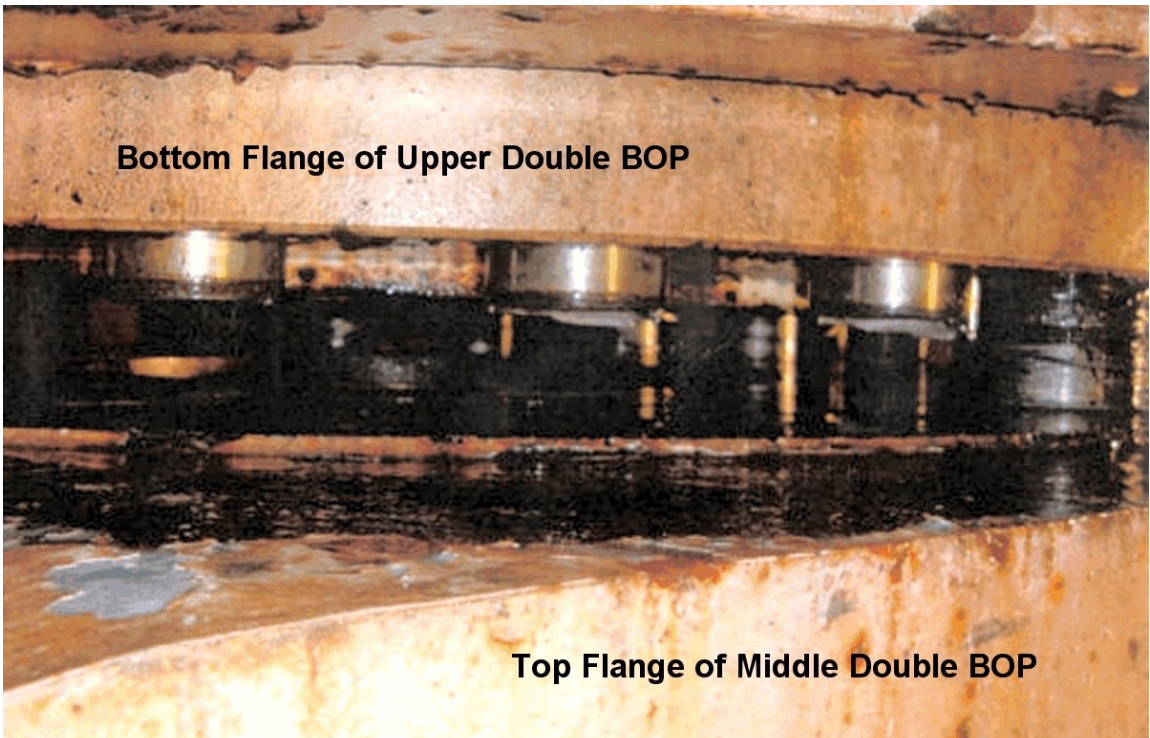


Figure 1

Source: DODI

View of fractured studs shortly after the failure.

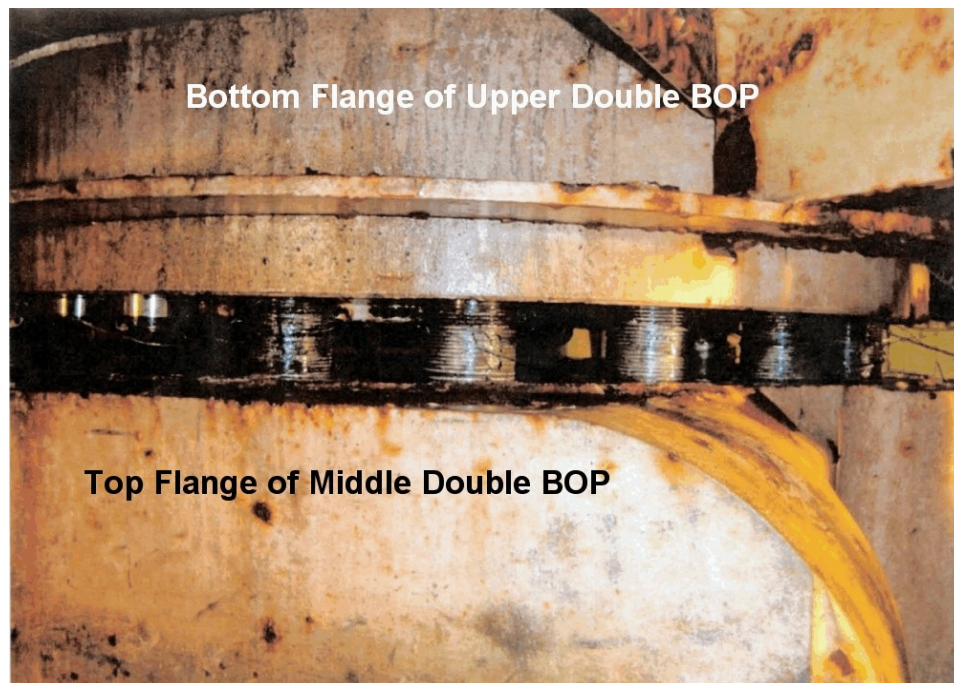


Figure 2

Source: DODI

View of studs with stripped threads shortly after the failure.

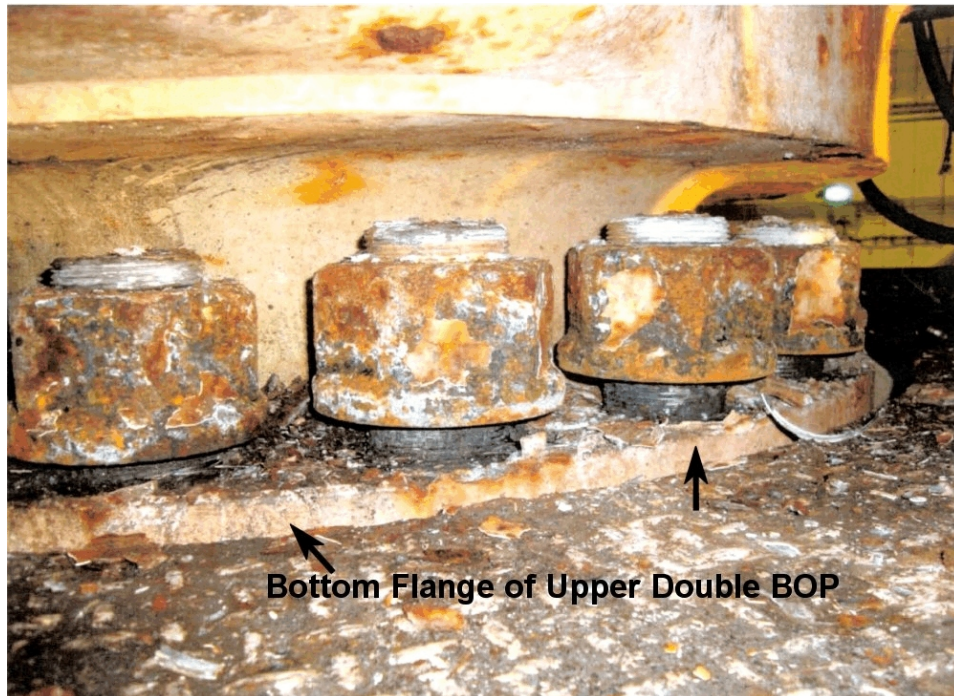


Figure 3

Source: DODI

View of the nuts on the top ends of the studs on the top side of the bottom flange of the upper double BOP, before they were cut to remove the failed studs.



Figure 4

Source: DODI

View of the top flange of the middle BOP showing the ends of fractured Studs 10 through 20, before removal.



Figure 5

Source: DODI

View of the nine studs that pulled out of the top flange of the middle BOP, stripping the female threads.



Figure 6

View of failed Studs 14, 15 and 16, three nuts and a new stud, as received.



Figure 7

View of the upper fracture surface on Stud 14. The white arrow indicates the apparent fracture origin location. The red arrow indicates the location of the cracked threads shown in Figure 11.



Figure 8

View of the lower fracture surface on the short section of Stud 14. The red arrow indicates the location of the cracked threads shown in Figure 11. The square hole was made to extract the stud section from the top flange of the middle BOP.



Figure 9

View of the upper fracture surface on Stud 15. The white arrow indicates the apparent fracture origin location.



Figure 10

View of the upper fracture surface on Stud 16. The white arrow indicates the apparent fracture origin location.

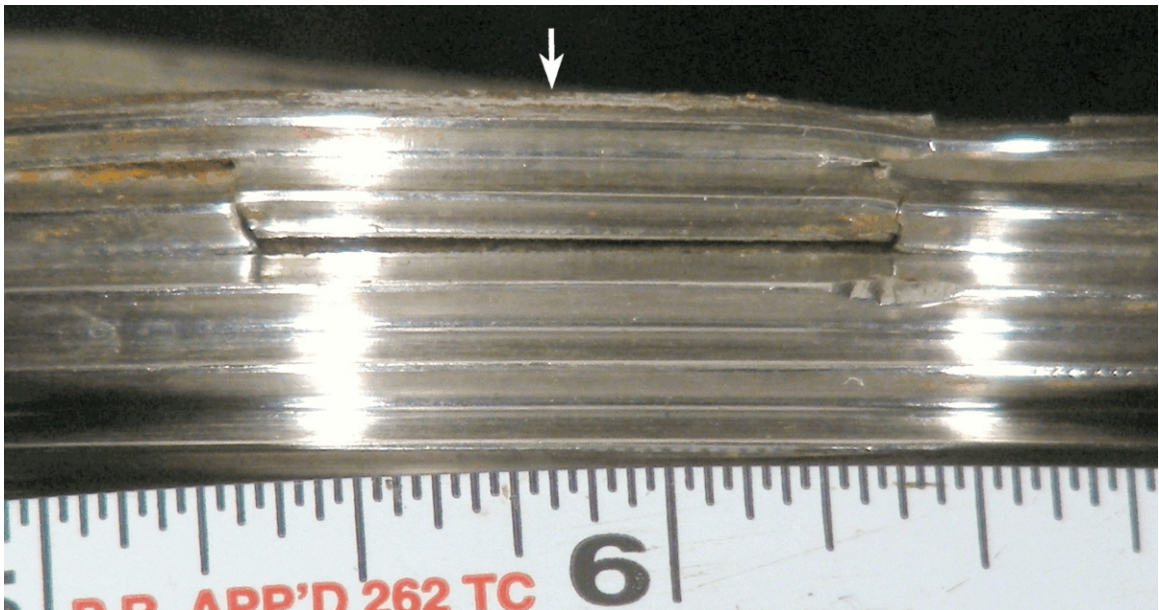


Figure 11

View of cracked threads on Stud 14, at the location indicated in Figures 7 and 8. The arrow indicates the location where a metallographic specimen was subsequently taken.

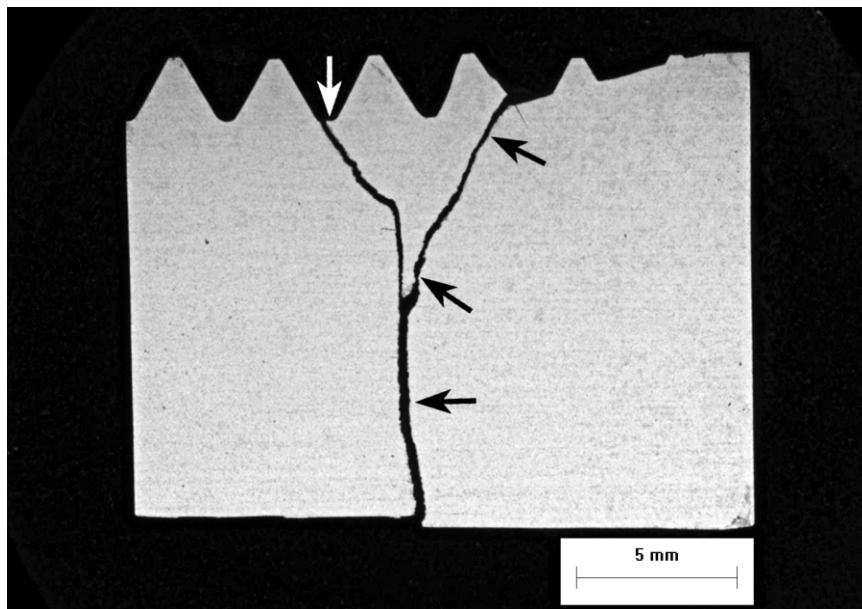


Figure 12 Kalling's No. 2 Etch Approx. 4X

Photomicrograph of the section of the cracked threads shown in Figure 11. A section of the fracture surfaces shown in Figures 7 and 8, is indicated by the black arrows. The cracked thread shown in Figure 11, is indicated by a white arrow.

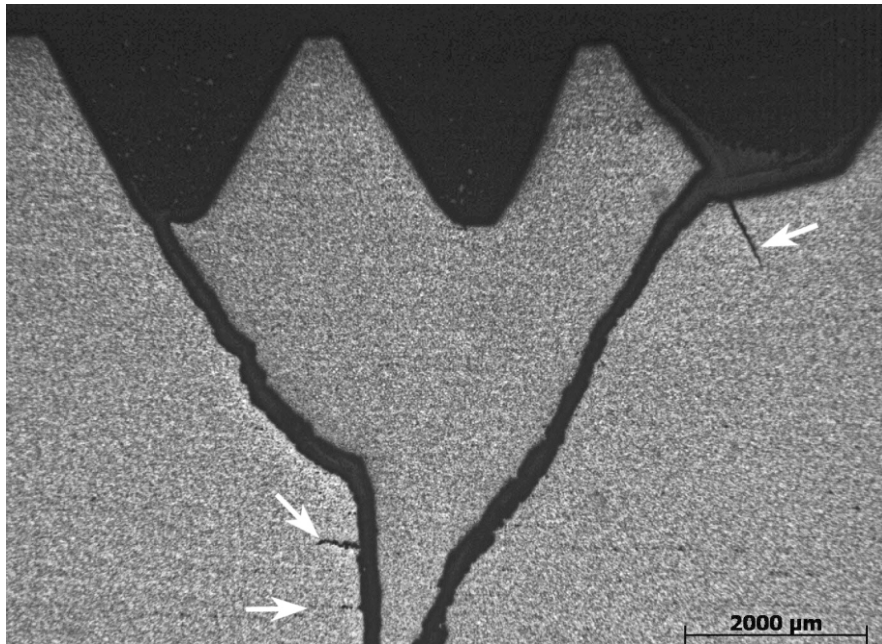


Figure 13 Kalling's No. 2 Etch 12X

Photomicrograph of a higher magnification view of the cracked threads showing small cracks, indicated by arrows.

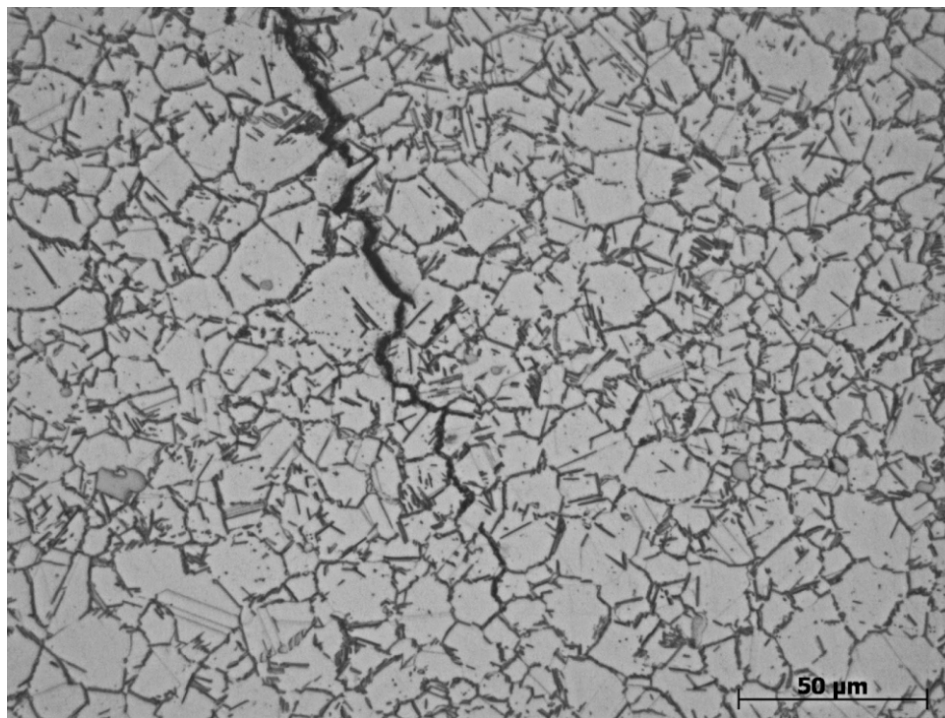


Figure 14 Kalling's No. 2 Etch 500X

Photomicrograph showing the tip of the upper right hand crack shown in Figure 13. Acicular δ -phase is shown along the grain boundaries and at intragranular locations.

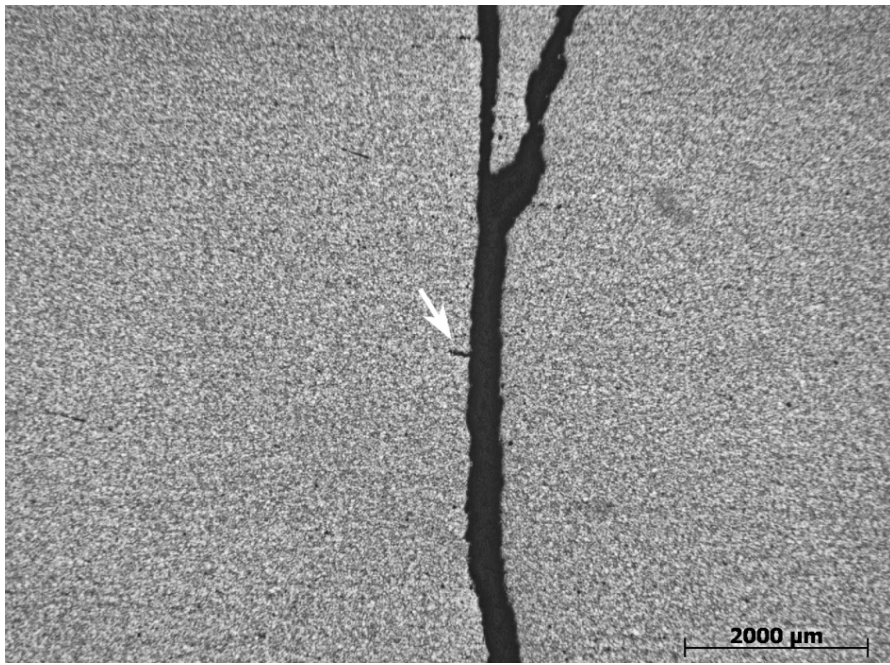


Figure 15 Kalling's No. 2 Etch 12X

Photomicrograph showing a small longitudinal crack, indicated by an arrow, originating at the transverse fracture surface.

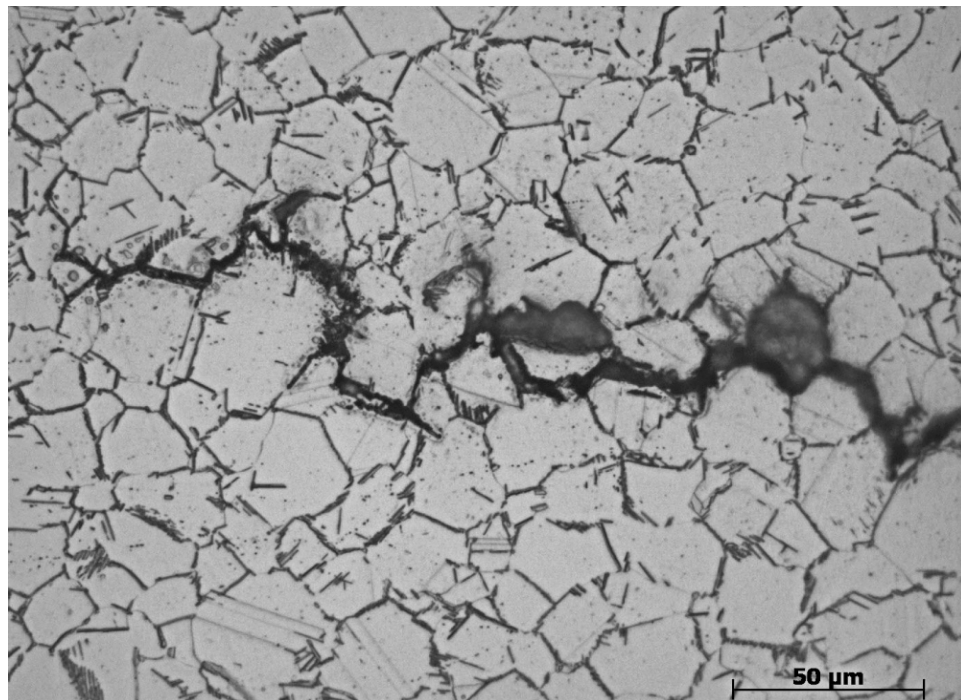


Figure 16 Kalling's No. 2 Etch 500X

Photomicrograph showing the tip of the small crack indicated by the arrow in Figure 15. Acicular δ -phase is shown intragranular and along the grain boundaries.

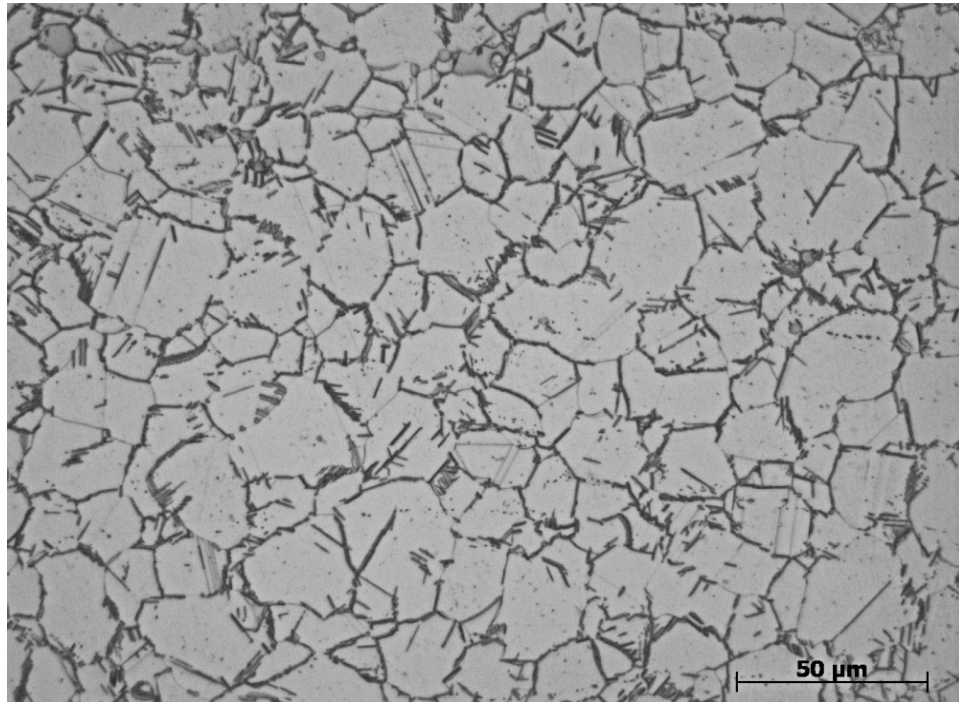


Figure 17 Kalling's No. 2 Etch 500X

Representative microstructure of Stud 14, showing the presence of acicular δ -phase along the grain boundaries and through grains.