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FAILURE ANALYSIS OF GRADE 12.9 BOLTS

EXAMPLE REPORT

Modified from Original Report

- Electronic Copy -

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SUMMARY

Bolt failure had been due to hydrogen embrittlement. High strength fasteners such as Grade 12.9 bolts, with hardness values over 39 HRC, are susceptible to hydrogen embrittlement. Hydrogen introduced by either acid pickling or zinc plating process had caused hydrogen embrittlement days or weeks after installation.

Proper baking after plating is the only method of ensuring reliability of plated, high strength bolts with hardness values of 39 HRC.

1.0 INTRODUCTION

[redacted] submitted seven failed ISO 4762 M6x25mm zinc-plated Grade 12.9 bolts for evaluation. The bolts were part of a recently installed linear rail assembly on a marine shaft lathe, which used one hundred bolts per rail. The bolts were observed to have failed several days after installation and prior to any significant rail use. Also included for evaluation were five good bolts, which had been located on either side of the failed bolts and remained unaffected.

Steel Image was requested to determine the nature of bolt failure.

2.0 EXAMINATION

Figure 1 displays the twelve bolts, comprising of seven failed and five good bolts, submitted for analysis. Fracture of the failed bolts had all occurred between the bolt head and shank (**Figure 2**). Stereoscopic examination found the fracture surfaces to be shiny and lacking in deformation, typical of brittle fracture. Some of the heads demonstrated a shear lip, associated with final failure.

Failed Bolt #1 was cleaned using an ultrasonic cleaner and prepared for examination using a scanning electron microscope (SEM). The images are displayed in **Figure 3**. The fracture surface exhibited predominantly intergranular fracture features, associated with embrittlement of the material. Examination of the other failed bolt heads found them to exhibit the same majority intergranular fracture appearance.

Lab fracture tests of the Failed and Good bolts were performed in an attempt to replicate the fracture appearance under simple overload conditions. Repeated tests found the bolts to consistently demonstrate a ductile appearance (**Figure 4**). This indicated that the material was ductile and ruled out intergranular fracture had been caused tempered martensite embrittlement or liquid metal embrittlement.

Chemical analysis of Failed Bolt #1 was conducted in accordance with ASTM E1019, E1097 (modified) and E1479. The bolt was found to conform to the compositional requirements of ISO 4762 (**Table 1**).

No hydrogen was detected within Failed Bolt #1 (**Table 1**). Yet once the zinc-plating had been compromised, hydrogen may have diffused out. Testing of a Good Bolt found high levels of hydrogen (17ppm), more than enough to cause hydrogen embrittlement. As all of these bolts would have been plated at the same time, Failed Bolt #1 would also have had high levels of hydrogen embrittlement. Therefore, the cause of bolt failure was due to hydrogen embrittlement.

Table 1: Failed Bolt #1 Chemical Analysis Results

Composition (wt%)							
Sample	**C**	**Mn**	**Si**	**S**	**P**	**Mo**	**Cr**
ISO 4762	0.20-0.50	--	--	0.035 Max	0.035 Max	--	--
Failed #1	0.34	0.79	0.20	0.006	0.020	0.16	1.01
	Ni	**As**	**Se**	**Sb**	**Al**	**Te**	**H**
ISO 4762	--	--	--	--	--	--	--
Failed #1	0.01	<0.01	<0.01	<0.01	0.03	<0.01	<3ppm
Good	-	-	-	-	-	-	17ppm

Microhardness testing of Failed Bolt #1 and Good Bolt #1 was conducted in accordance with ASTM E384 using a 500gf load. **Table 2** summarizes the obtained results. Both the Failed and Good Bolts conformed to the hardness requirements. Note that Grade 12.9 bolts had hardness values over 39 HRC which leave them susceptible to hydrogen embrittlement.

Table 2: Core Microhardness Test Results

Location	**Measurements (HV_{500gf})**	**Avg. Hardness**	
		HV_{500gf}	**HRC**
Dwg Req.		385-435	39-44
Failed Bolt #1	419, 442, 439, 431, 436	433	44
Good Bolt #1	422, 431, 429, 432, 436	430	44

Cross-section were taken through Failed Bolt #1 and Good Bolt #1 and prepared for optical examination in accordance with ASTM E3. The images are displayed in **Figure 5 and 6**. Overall the crack path was linear with no branching. No quality issues or concerns were observed with the bolt material. Both the Failed and Reference bolts displayed a martensitic microstructure typical for a quench and tempered alloy steel.

3.0 CONCLUSIONS

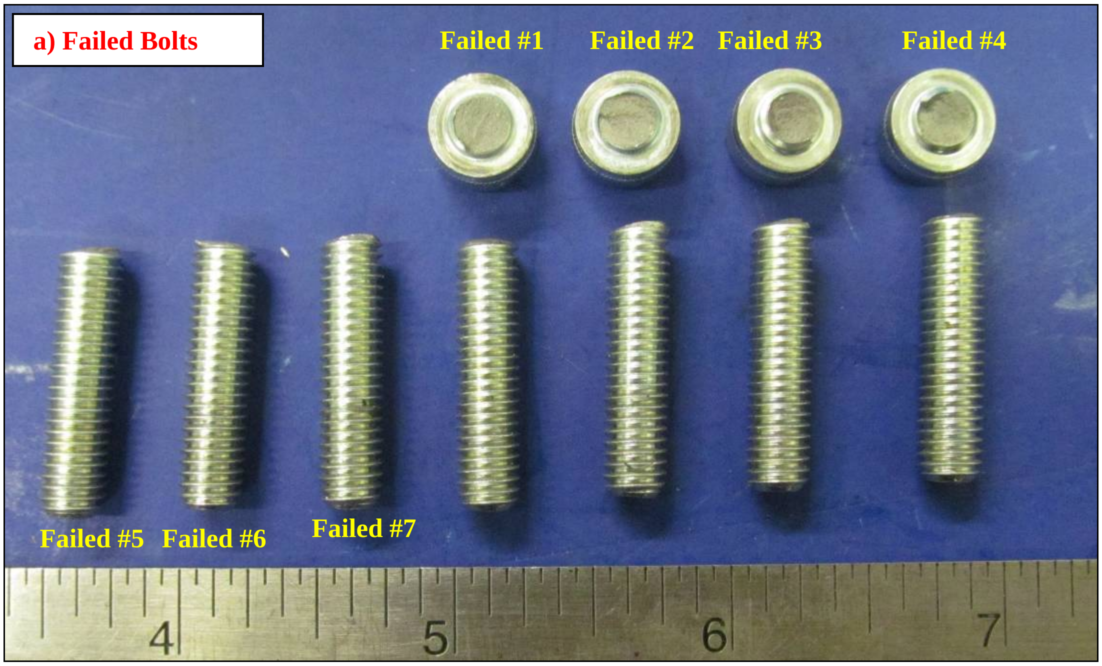
Bolt failure was due to hydrogen embrittlement. High strength fasteners such as Grade 12.9 bolts, with hardness values over 39 HRC, are susceptible to hydrogen embrittlement. Hydrogen introduced during either acid pickling or zinc plating process had caused hydrogen embrittlement and intergranular fracture days or weeks after installation.

Even low levels of hydrogen within high strength steel, at levels above 3ppm, can cause hydrogen embrittlement delayed cracking. Therefore, hydrogen levels of 17ppm in a good bolt, from the same bolt lot as the failed bolts, indicated that ample hydrogen had been present for hydrogen embrittlement. Combined with the intergranular fracture features and lab-fracture illustrating ample ductility, bolt failure was diagnosed as hydrogen embrittlement.

Although no hydrogen was detected within the failed bolt tested, this may have been due to hydrogen having been able to diffuse out after fracture. Hydrogen introduced during either acid pickling or zinc plating is retained/trapped within the bolts by the zinc-plating. Therefore, the unplated fracture surface would have provided a diffusion path for hydrogen to escape from.

Plated Grade 12.9 bolts require a proper bake-out to remove hydrogen introduced during acid cleaning and plating. Proper baking is the only method of ensuring reliability of plated, high strength bolts with hardness values of 39 HRC. ASTM F1941 recommends baking for 14-24 hours at 374°F to 428°F (190-220°C) for bolts with a hardness of ~44 HRC.

Other bolts in this lot would also be at risk of hydrogen embrittlement failure.



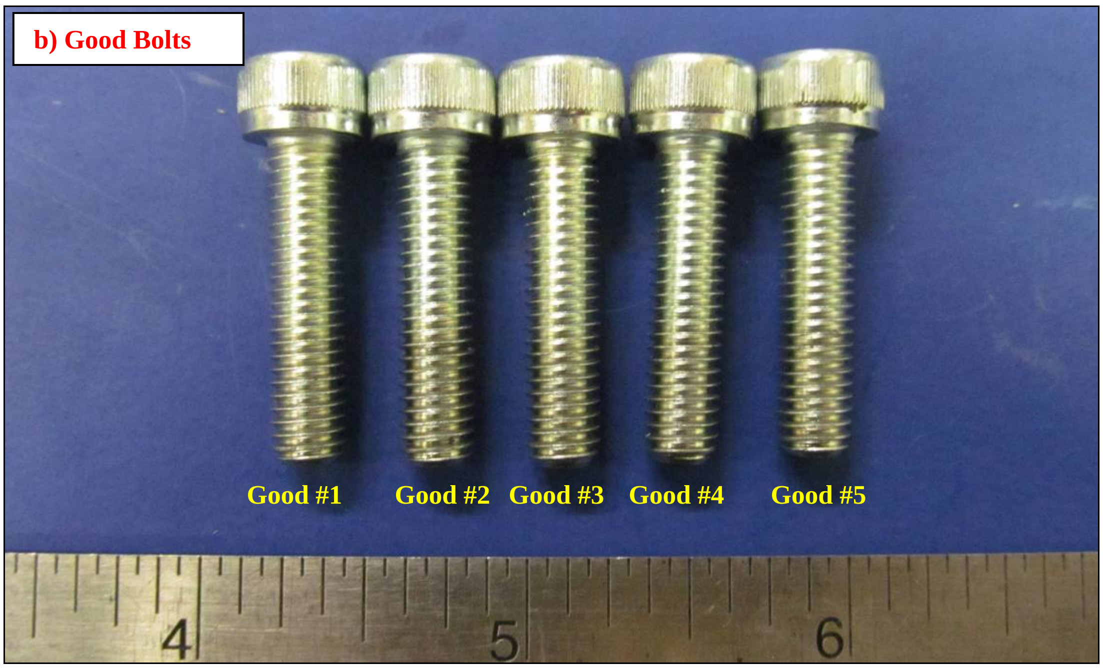


Figure 1: Photographs displaying the twelve bolt samples submitted for analysis. These included parts of seven failed bolts and five good bolts, which had been located on either side of the failed bolts during assembly.

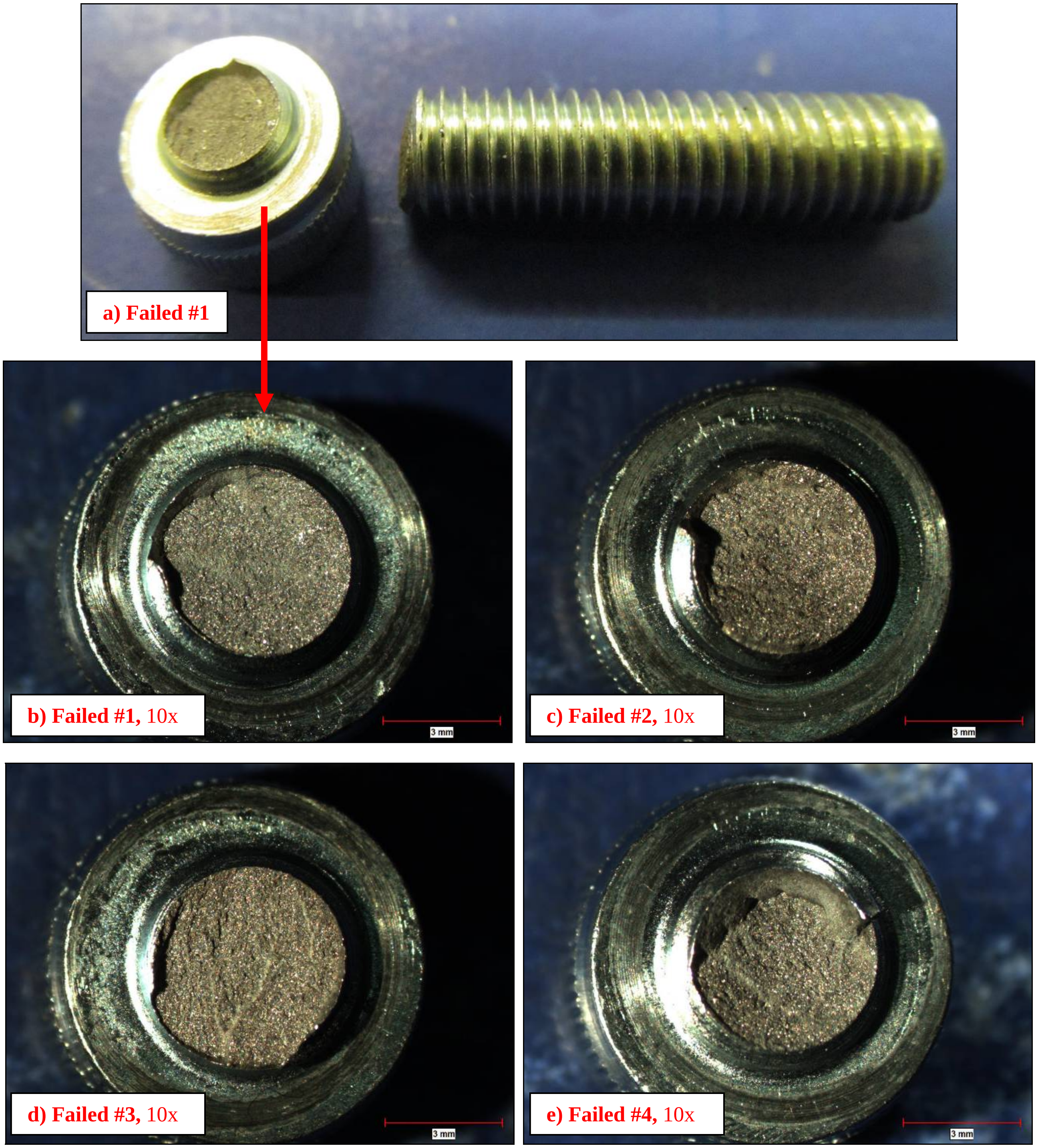


Figure 2: Photograph and macrographs displaying the four failed bolt heads. All four exhibited shiny, crystalline fracture features void of deformation.

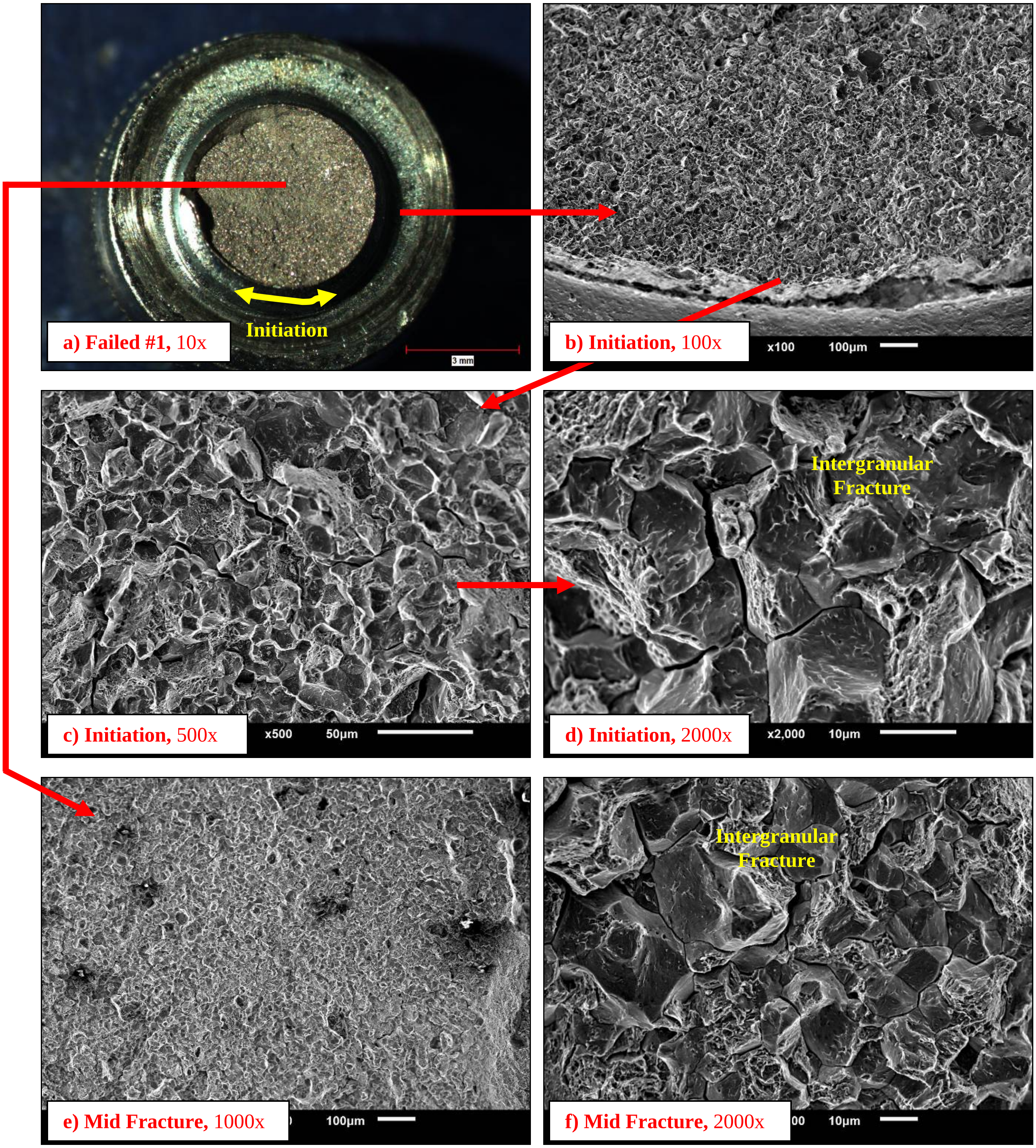


Figure 3: Macrograph and SEM images of the fracture surface of Failed Bolt #1. The entirety of the fracture comprised of a mixed intergranular/microvoid failure mode indicating failure had occurred due to an embrittling phenomenon (later confirmed to be hydrogen embrittlement). SE1, 15kV.

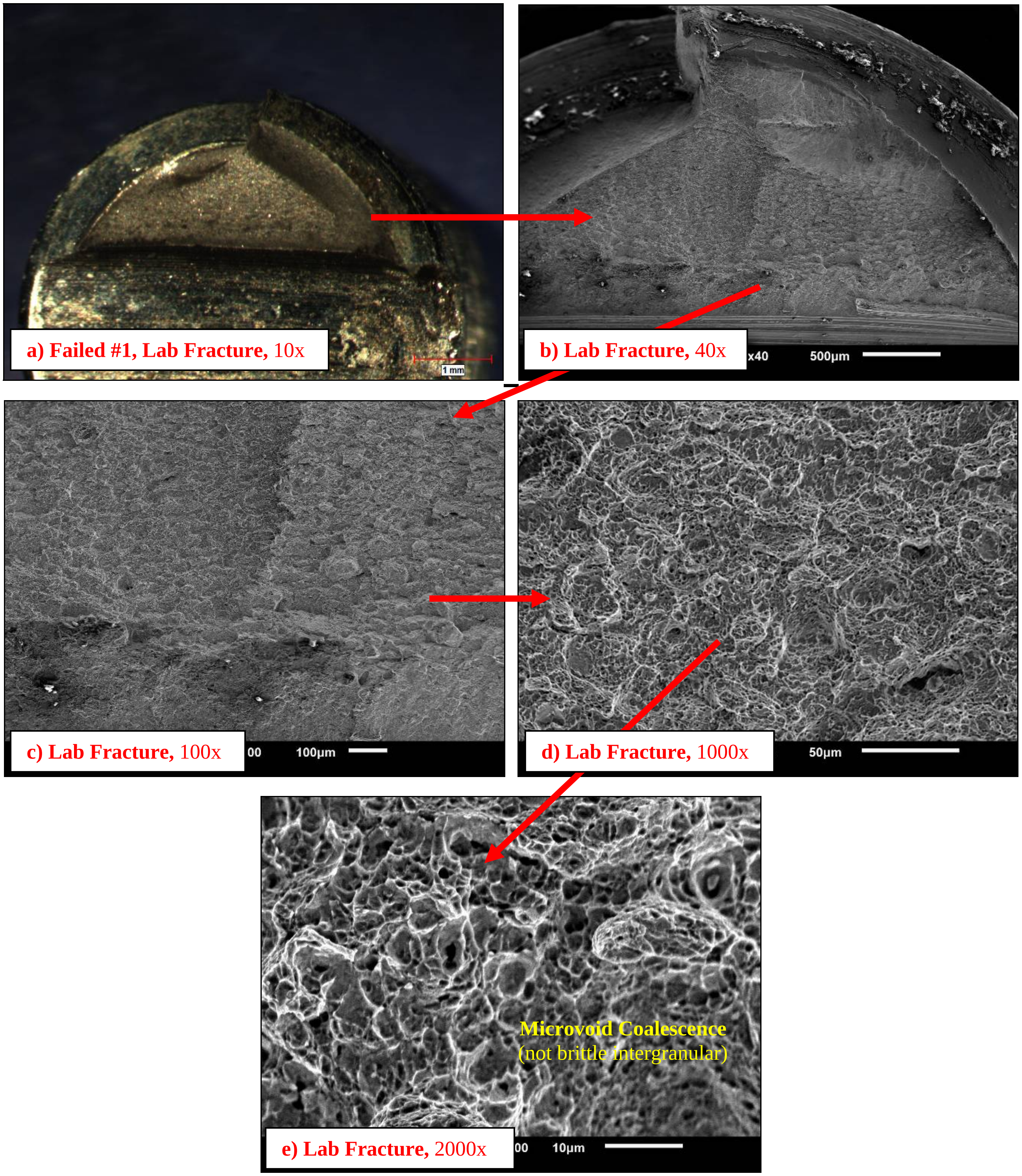


Figure 4: Macrograph and SEM images of the fracture surface of the lab fractured Failed Bolt #1. The entirety of the fracture exhibited microvoid coalescence fracture features consistent with ductile overload and quite different from the actual fractures under investigation. Therefore, the material was ductile under overload conditions. SE1, 15kV.

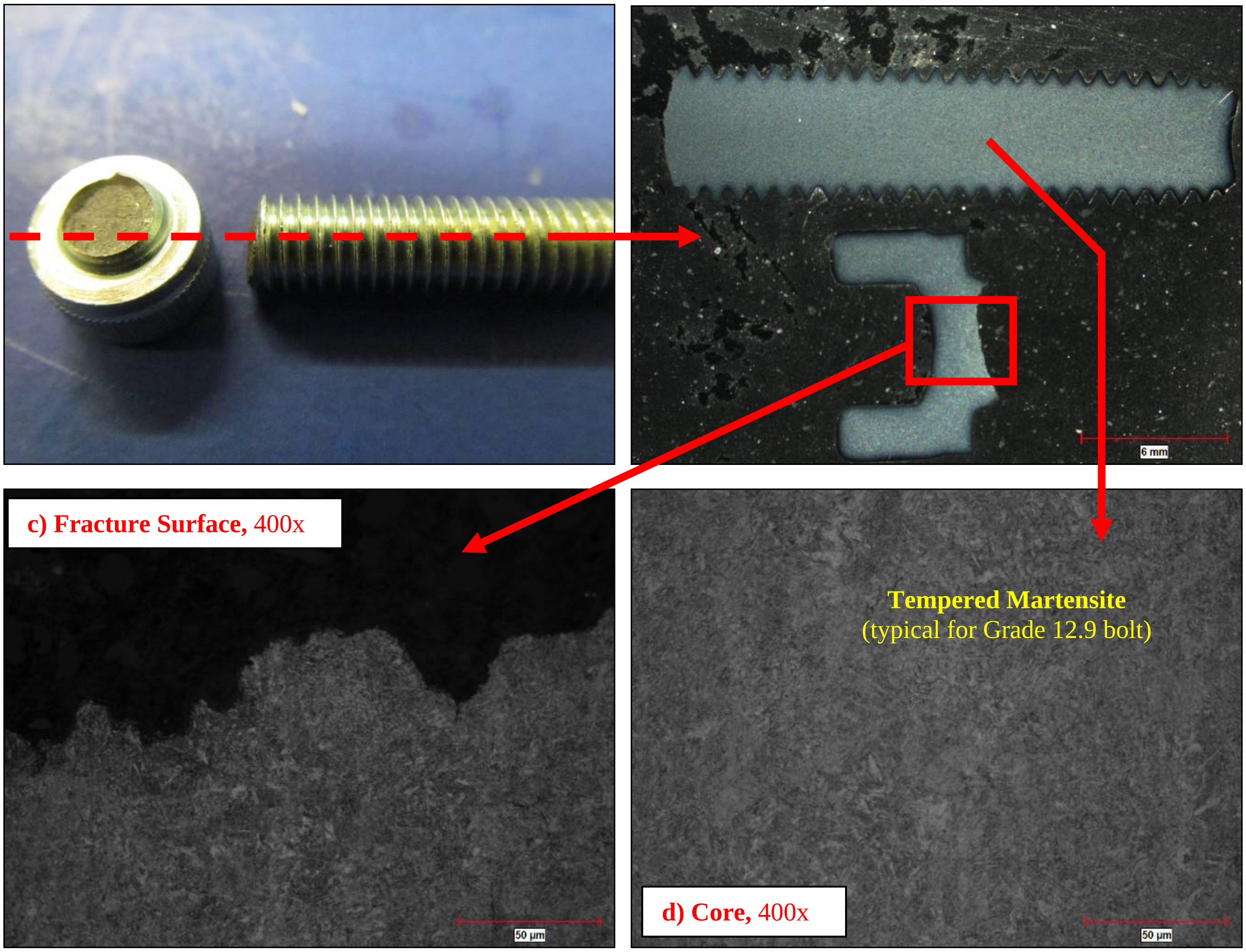


Figure 5: Photograph, macrograph and micrographs displaying the cross-section taken through Failed Bolt #1. The fracture did not feature any branching or secondary cracking. The core structure comprised of tempered martensite typical for Grade 12.9 bolts. No quality issues or concerns were observed in the fracture region by optical microscopy. Etched using 3% nital.