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1. Introduction

Failure analysis is a valuable tool improving quality in material selection and mechanical design; learning from failure one can act correctively, eliminating these causes, or preventively, preventing them from occurring and minimizing accidents and financial losses [1,2]. Screws failure typically came from fatigue damage, overload, corrosion or hydrogen embrittlement, or combinations of all these failures [3,4].

Screws are used in nonpermanent fasteners, since most machine parts that require connection must often be made in such a way that they can easily disassembled as well as assembled. In general, there is a preload applied, especially in applications involving fatigue loading [5–7]. Steel is the material of which most fasteners are made [5]. Low-carbon, medium-carbon, and alloy steel grades are used to make all various strength grades and property classes of threaded steel

fasteners suitable for service between -50°C and 200°C . When screw section size increase, hardenability becomes an important factor in materials selection for screw fabrication [8].

The choice of material depends on many factors, including its corrosion behavior. Corrosion is the degradation of a metal by an electrochemical reaction with the environment. The most common method of preventing corrosion is the selection of the metal or alloy for a particular corrosive service. Other important considerations are the geometry and configuration of the joint, presence of passive or active coatings, anodic or cathodic inhibitors, cathodic protection or even monitoring that involves preventive maintenance and regular replacement. In many practical applications, the contact of dissimilar materials is unavoidable, and a potential difference usually exists between two dissimilar metals when they are immersed in a corrosive or conductive solution. If these metals are in electrical contact, potential difference produces electron flow between them. The less resistant metal becomes anodic and the more resistant metal cathodic. On ferrous metal screws, the most common protection is the use of protective metal coating, in general, zinc, cadmium, or aluminum [9–11].

This paper presents a case study of the failure analysis on M42 screws used in a power generator, including background information, visual examination, material properties determination, fractographic and metallographic examination and engineering analysis, pointing and describing the mechanisms involved in the failure cause and proposing solutions to mitigate this problem.

2. Background

To determine fracture cause, power generator M42 screws were received for analysis. Limited information was available regarding service time, however the failure was premature and unexpected. The screws are from a power generator that operates in unfavorable environmental conditions, exposed to rainwater and residues that can lead to sulfuric acid solution formation.

3. Experimental analysis

On visual examination, preliminary observations indicated a failure linked to stress and corrosion simultaneously. The screws exhibit corrosion residues, fully brittle fracture macroscopic appearance, crack nucleation at concordance radius between the screw body and head, and fracture surfaces showed advanced corrosion (Fig. 1). It should be noted surface protection by electrochemical treatment and red paint.

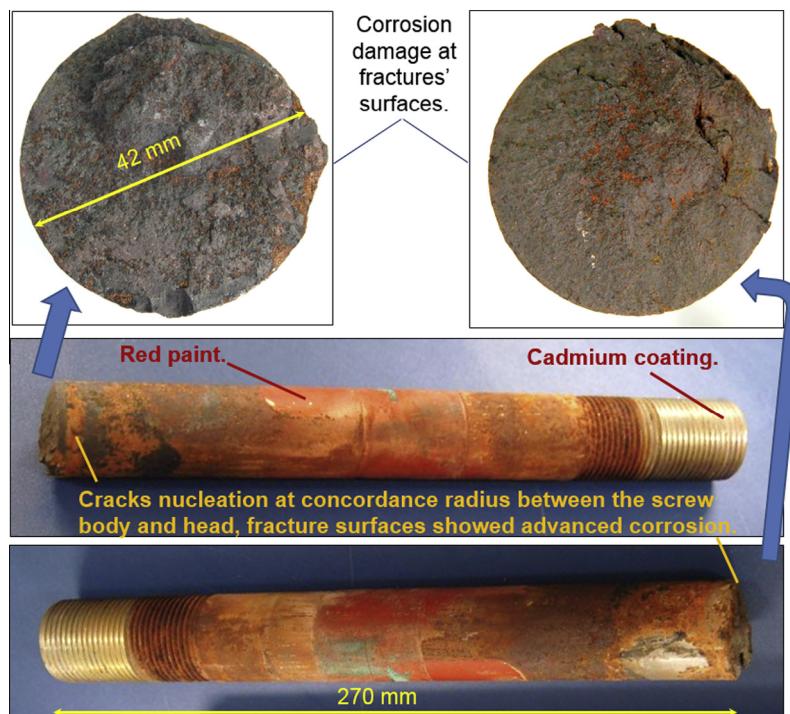


Fig. 1. M42 screws fractures samples. The arrows point fracture sections. The screws exhibit red corrosion residues and fully brittle fracture macroscopic appearance. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

Table 1
Product analysis element (% by weight).

	C	Cr	Mn	Mo	B	Ni	P	S	Si
Specimen	0.38	0.76	0.66	0.22	...	2.07	0.015	0.015	0.20
ASTM F568M-07 class 8.8	0.25–0.55	Max 0.048	Max 0.058	...

Table 2
Mechanical properties.

	Yield strength, MPa	Tensile strength, MPa	Elongation, %	Reduction of area, %	Rockwell hardness
Specimen ASTM F568M-07 class 8.8	913 Min 660	1030 Min 830	19 Min 12	55 Min 35	C33.8 C23–C34

From material properties determination, chemical, hardness and tension analysis were carried out to confirm material specification. **Table 1** show chemical analysis results performed according ASTM A751 – 11 on inductively coupled plasma mass spectrometry (ICP-MS) and by combustion (LECO) [12]. The screw material is similar to an AISI 4340 low alloy steel and shall conform to ASTM F568M – 07 composition specifications [13].

Table 2 show tension test results according ASTM E8/E8M – 13a [14] performed on MTS 810 material testing system (Fig. 2a) and engineering stress-strain curve can be seen in Fig. 2b. The hardness Rockwell scale, measured in the cross section profile, according ASTM E18 – 14 [15] is showed on (Fig. 2d). From **Table 2**, mechanical properties showed that results are consistent to class 8.8 screws resistance, according to ASTM F568M – 07 [13]. This specification covers chemical and mechanical requirements for nine property classes of carbon and alloy steel externally threaded metric fasteners in nominal

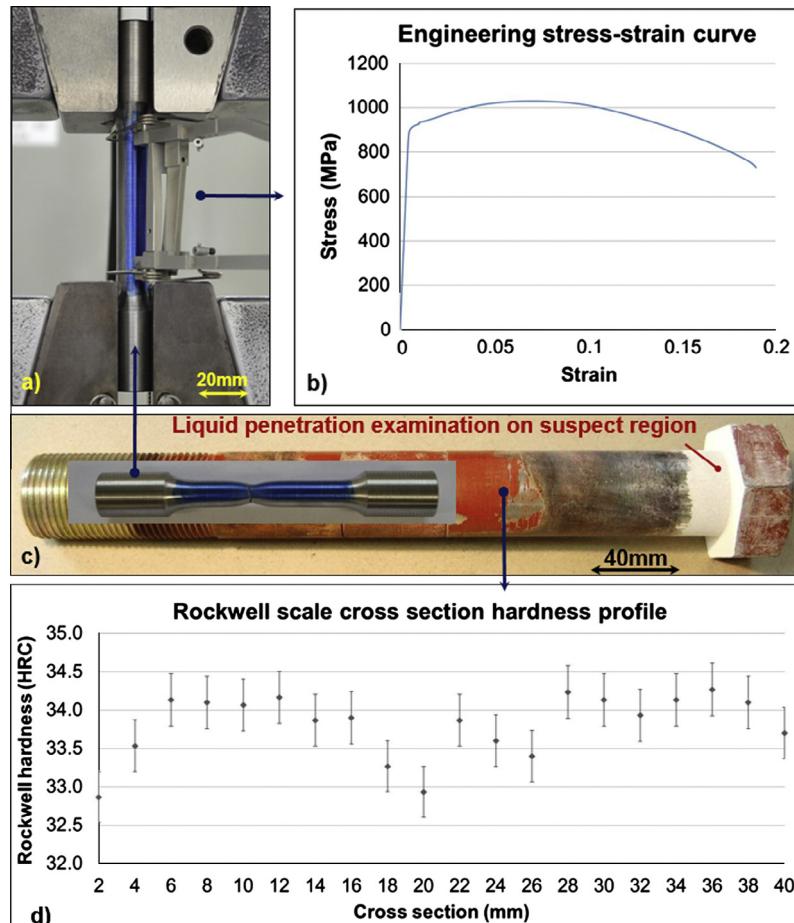


Fig. 2. (a, b and c) Tension test position, engineering stress–strain curve and preexisting cracks on components tensile tests; (d) hardness profile.

thread diameters M1.6 through M100 suited for use in general engineering applications, and it was adopted as compatible component specification, as it was not supplied a screw drawing or another specification.

On fractographic examination, due to advanced fracture surface corrosion and handling mechanical damage, it was not possible to see details by visual analysis, however it is worth noting that there is not considerable plastic deformation. However, fracture detail observation is imperative to determine failure cause, and the solution was to look cracks in other not fractured screws. For this purpose, non-destructive tests according ASTM E165M – 12 Liquid Penetrant Examination [16] were performed but, unfortunately, it was not found significant crack evidence ([Fig. 2c](#)).

With the aim to verified fracture aspect, tensile tests on screws which did not fracture in service was carried out. It was necessary to reduce the screw cross section area through a central hole, allowing the available load equipment to break the sample. Fresh produced fractures showed undamaged preexisting cracks ([Fig. 3](#)), suitable to be subjected to scanning electron microscope (SEM) analysis, carried out in a CamScan CS 3200 LV. It was observed the occurrence of intergranular fracture, with gaps between grains on preexisting cracks region and dimples on no cracks location ([Fig. 4a and b](#)).

Metallographic examination showed tempered martensitic microstructure, compatible to ASTM F568M – 07 standard specifications [16]. Surface fracture longitudinal section presents substantial degradation by intergranular cracks ([Fig. 4c](#)) and corrosion products inside the crack ([Fig. 4d](#)); this fact can explain no significant crack evidence at liquid penetrant testing: under loading of the screw, the crack is open, but when unloaded it automatically closes, and the presence of oxides also help closing the crack to the liquid used in the testing. Coating surface analysis showed that average coating thickness is 8.44 μm , and its composition is predominantly cadmium, determined by energy dispersive spectroscopy (EDS-SEM). Better condition preserved surface regions were observed ([Fig. 5a](#)), however, there are localized corrosion points ([Fig. 5b](#)). It can be seen that the coating was preferentially corroded where the paint is faulty ([Fig. 5c](#)).

EDS analysis on residues located within the cracks indicated that they are predominantly composed by iron, nickel, cadmium and oxygen ([Fig. 6a](#)), confirming the hypothesis that this is a corrosion product. The backscattered electrons image reinforced the observation of intergranular aspect of corrosion ([Fig. 6b and c](#)).

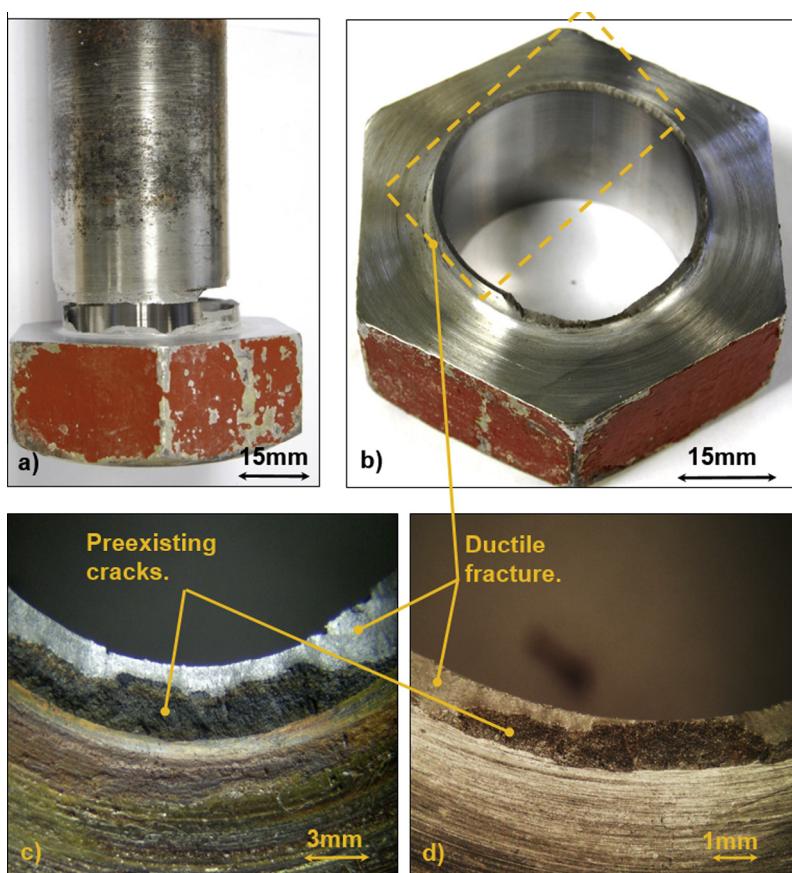


Fig. 3. Preexisting cracks on components tensile tests; (a) fracture aspect on component tensile test; (b) region without preexisting cracks; (c and d) detail of preexisting cracks and ductile fracture.

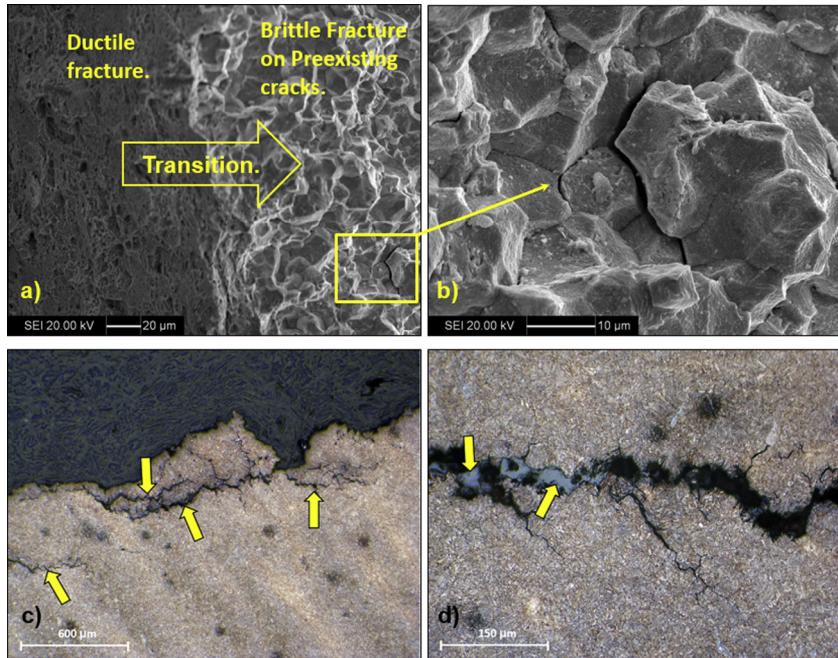


Fig. 4. (a and b) Scanning Electron Microscopy (SEM) – Secondary Electron Images; (a) ductile to brittle transition and grains gaps; (b) grain gaps in detail. (c and d) optical microscopy (OM) after Nital 2% etching showing tempered martensite and (c) large degradation by cracks and (d) residues inside the crack.

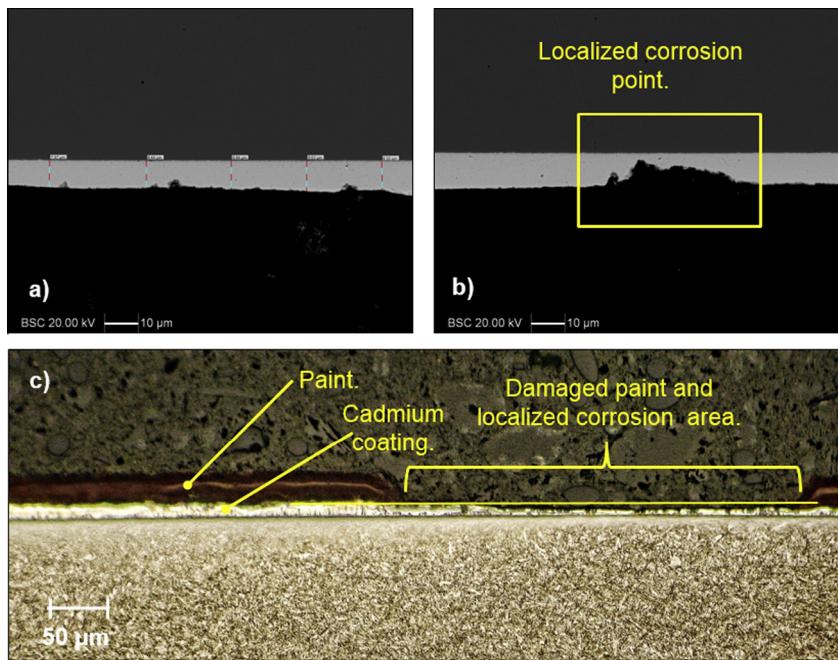


Fig. 5. (SEM) – Backscattered Electron Images; (a) better condition preserved coating regions and measurements; (b) localized corrosion point and (c) corrosion area detail where the paint is damaged (OM).

4. Engineering analysis

On joining processes, screws are used to fasten together various assembly parts, and to prevent assembly release, a tensile force greater than the force tending to separate the parts must be applied to the screw. The use of sufficient initial tension is very advantageous in reducing the fatigue effects in the screw if load is not steady but fluctuating. On dynamic applications,

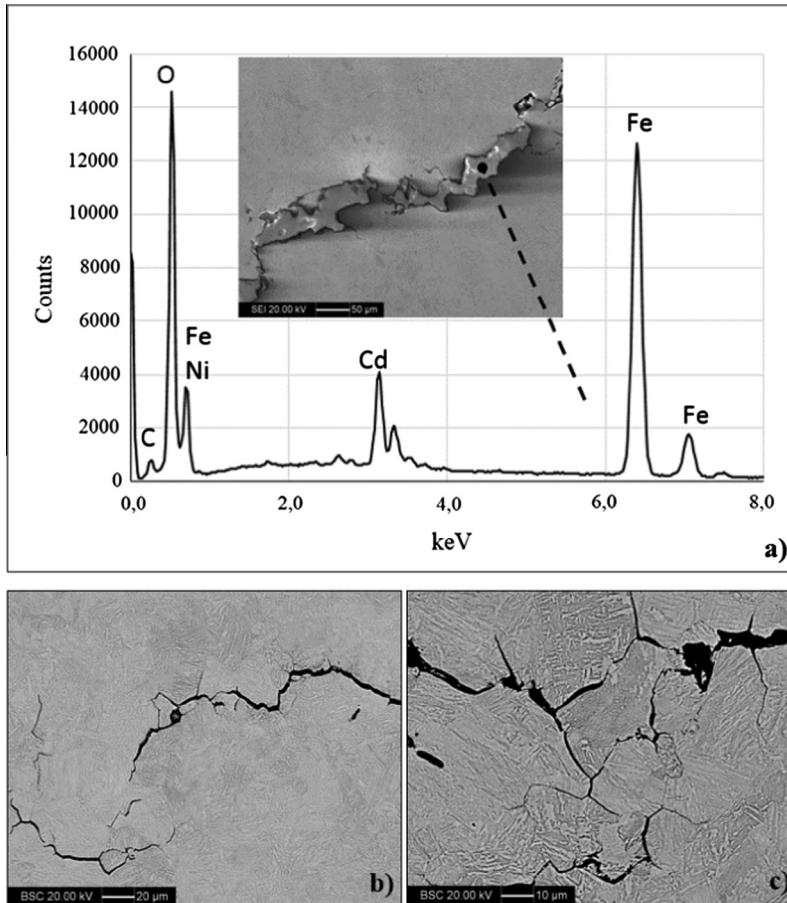


Fig. 6. (SEM); (a) EDS in residues on the cracks; (b and c) Backscattered Electron Images, intergranular cracks aspect observation.

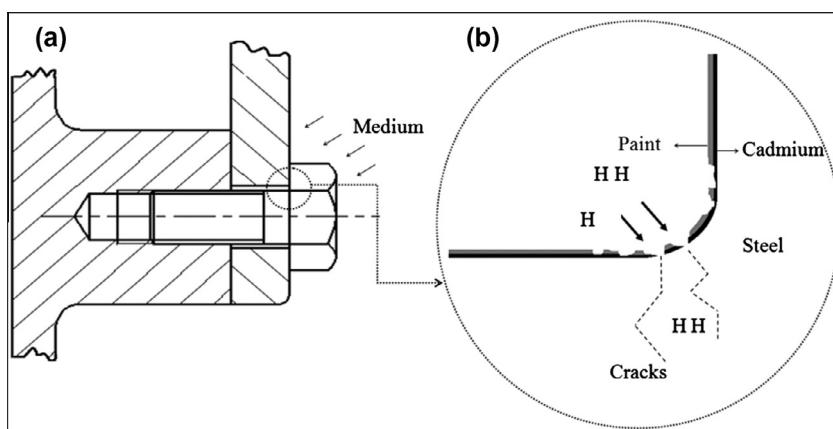


Fig. 7. Failure mechanism; (a) screw assembly schematic representation; (b) zoom region of (HSC) cracks initiation.

screw load should retain a compressive part load range variation and minimize tensile screw force range variation, thereby, fatigue effects, which depend largely on the extent of the variation of stress, are correspondingly reduced [5–7]. Thus, it is natural that screws are undergoing high stresses (lower than the material yield strength) in their engineering applications.

Considering that screw material is in conform with the typical ASTM F568M – 07 standard specifications [13], the failure source was not material selection and heat treatment stages, despite the fact that two hardness points were below 33 HRC (both presented 32.9 HRC); scatter in hardness values was small and mean hardness value was 33.8 HRC. Visual fracture

examination indicated no plastic deformation, and this fact is incompatible with the observed mechanical behavior (19% elongation and 55% necking) if the failure cause was overload.

From background information, sulfur acid solution could be present on environment. Stress, sulfur acid solution environment and martensitic carbon steel is a combination that allows to focus on a corrosion failure.

SEM fractographic analysis showed intergranular fracture with gaps between grains on preexisting cracks region. Other researchers have pointed out this type of fracture due to hydrogen embrittlement (HE) [1,17,18] simultaneously with assembly stress or stress concentration, naming it hydrogen stress cracking (HSC) [1]. HSC failure mode is usually characterized by failure along grain boundaries, and can be explained by hydrogen-enhanced de-cohesion (HEDE) mechanism, where monoatomic hydrogen travels to a region of high triaxial stress resulting in crack propagation along the metallurgical discontinuities, preferably toward grain boundaries [1,18]. Hydrogen stress cracking (HSC) is related to the brittle fracture of a normally ductile alloy under a substantial load in the presence of hydrogen. Carbon and low-alloy steels, stainless steels, nickel alloys, and aluminum alloys are susceptible to HSC. Hydrogen stress cracking is also referred to as hydrogen-induced cracking (HIC), hydrogen-assisted cracking (HAC), delayed fracture and static fatigue. In the absence of a sharp initial crack, the hydrogen-induced fracture often starts at subsurface sites where triaxial stress is highest. If a sharp crack is present, the hydrogen cracking may start at the tip of the preexisting crack. High hydrogen concentrations ahead of the crack tip help the crack grow [10].

One hydrogen source could be the cadmium coating performed on electrodeposited cadmium process, where hydrogen is produced due to hydrogen partial cathodic reaction [18,19]. Although there is no information available of baking treatment after the electrodeposition of cadmium before the paint process, observations such as the presence of screws without any defect in equipment assembly on dry areas, the fact that on electrodeposition process the hydrogen is available across the entire screw surface, and the ductile fracture regions on the surface after the tensile test (Fig. 3b), it could be concluded that hydrogen entered in the material by a localized manner and thus, electroplating treatment was not a source of hydrogen.

Another source of hydrogen could be the service conditions, where the less resistant metal becomes anodic and the more resistant metal cathodic, since they are in an electrical contact in a corrosive environment. The anodic reaction is the oxidation of a metal to its ion, and in the context of this study, $\text{Cd} \rightarrow \text{Cd}^{+2} + 2e$, and the cathodic reaction is $\text{H}^+ + e \rightarrow \text{H}$. In galvanic corrosion the area effect is an important factor. For a giving current flow in the cell, the current density is greater for a small electrode than for a larger one. The greater the current density at an anodic area, the greater is the corrosion rate [10,11].

In this case, one can consider cadmium coating as the anode and steel as the cathode. Thus, the steel protection depends on the uniform corrosion of the cadmium coating on environment.

If a localized part of steel is exposed to the environment, the cathodic reaction that produces hydrogen will occur only over the exposed steel, since cadmium corrosion process will occur in the larger area. Due to paint damage, small cadmium coating surface areas were exposed, accelerating corrosion rate and exposing the steel. The cadmium content on corrosion product inside the crack strongly suggest anodic dissolution of cadmium coating corroborating the hypothesis of service conditions as the hydrogen source.

An inspection on least corroded areas, near the fracture and cracks, realizes that on these areas, where the cadmium coating was localized corroded, paint damage can be found.

This evidence suggests that a small cadmium coating area was exposed to the environment, thus, undergoing accelerated corrosion and exposing the steel. The steel became the cathode and the cadmium corrosion produced hydrogen that diffused to the steel grain boundaries. After all exposed cadmium in the paint damaged areas was corroded, the anodic protection given by the coating was lost, and corrosion of the steel is also possible. This can be the reason for the presence of both cadmium (from coating) and iron and nickel (from steel) in the oxide corrosion products (Fig. 6).

However, it also was observed corrosion product within intergranular cracks of screw under sustained assembly load, this may suggest stress corrosion cracks (SCC), where crack morphology can be intergranular and transgranular, and on the alloy surface the appearance usually is little corroded [10,11]. It is suggested that SCC mechanism is the anodic dissolution, where metal is corroded on crack tip [20,21], on the other hand, changes on the environment and localized chemical reaction on corroded crack tip can produce a cathodic reaction and allows hydrogen embrittlement (HE) to simultaneously occur to SCC [22].

Thus, due the pronounced surface corrosion and cadmium coated leaching, predominant intergranular crack and absence of corrosion between grain boundaries on fresh produced fractures, it is believed that component failure occurred initially by hydrogen stress cracking (HSC) predominantly, after that, open cracks surfaces on loaded screws became subject to corrosive environment causing corrosion products, at this point we can have a concurrent corosions processes like HSC and SCC.

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1 Introduction

Screws have a crucial role in various industries by facilitating the temporary joining of multiple components. Failures in such fasteners can result in severe structural damage. This report aims to condense a specific article's findings that delved into a case study concerning the analysis of M42 screws utilized in power generators. These screws were collected from a power generator operating in unfavorable environmental conditions. The batch included both failed and non-failed screws, with the unexpected failures occurring in the former group.

The screws underwent multiple rounds of experimental analysis, followed by thorough engineering analysis of the experimental data to determine the causes and processes behind their failures. Chapter 2 provides an overview of the research team's experimental analyses, while Chapter 3 elaborates on the engineering analyses conducted on the experimental findings. Finally, Chapter 4 presents recommendations aimed at preventing similar failures in the future.

2 Investigation Methods

To begin their investigation, they conducted a visual examination, identifying a simultaneous connection between the failure and both stress and corrosion. The fracture appeared entirely brittle, and the point of crack initiation was observed at the radius where the screw body met the head. Moreover, the screws exhibited corrosion residues, and the fracture surface displayed clear indications of advanced corrosion.

Given the unavailability of precise information regarding the screw material, the research team conducted chemical, hardness, and tension analysis to ascertain the material properties. The chemical analysis revealed a similarity to AISI 4340 low alloy steel. Furthermore, the hardness and tensile tests confirmed that the screws met the resistance standards of class 8.8 screws. These findings aligned with the specifications outlined in ASTM F568M-07, a standard for carbon and alloy steel fasteners.

A fractographic examination of the screws was conducted, but it did not yield any significant results. Notably, no significant plastic deformation was observed during this examination. Additionally, a non-destructive examination called Liquid Penetrant Examination was performed to detect any signs of cracks, but no visible indications of cracks were observed.

Subsequently, they conducted a tensile test on intact screws that were in service but had not experienced fractures, aiming to investigate fracture characteristics. These fractures revealed the presence of undamaged preexisting cracks. The scanning electron microscope analysis of the fractures indicated that intergranular fractures had occurred, with gaps observed between the grains.

Lastly, the coating analysis revealed that the average coating thickness measured 8.44 µm, with cadmium being the predominant material in the coating. It was observed that the coating had undergone preferential corrosion in areas where the paint was faulty. Furthermore, an analysis of the residues found within the cracks showed a dominant composition of iron, nickel, cadmium, and oxygen. This discovery provided further confirmation that the failure was indeed linked to corrosion.

3 Failure Causes

The next phase involved interpreting the experimental results to pinpoint the primary cause of the failure. Based on the hardness and tensile tests conducted on the screws, they were able to confirm that the failure could not be attributed to either a material selection error or shortcomings in the heat treatment stages.

Based on the results obtained from SEM imaging, which revealed intergranular fractures with gaps between grains in the preexisting crack region, they attributed this phenomenon to hydrogen stress cracking (HSC). This failure mechanism can be explained by the movement of hydrogen molecules toward areas with high triaxial stress, ultimately causing cracks to propagate along metallurgical discontinuities. An important factor in this failure investigation was identifying the source of hydrogen. One potential source they explored was the cadmium coating process, which can produce hydrogen. However, their experimental observations did not support this hypothesis, leading them to conclude that the coating process could not be the source of the hydrogen.

Secondly, they considered the possibility that the service conditions could be a source of hydrogen. Specifically, they proposed that a combination of anodic and cathodic reactions could generate hydrogen. In the case of the screws, the anodic reaction occurred on the less resistant metal, namely the cadmium coating, while the cathodic reaction occurred on the more resistant metal, which is the steel core of the screws. Their observations revealed that even a small area of exposed cadmium coating could lead to corrosion, exposing the steel to the environment and cadmium. This, in turn, could initiate the anodic and cathodic reactions, resulting in the production of hydrogen.

Ultimately, the confirmation of corrosion residues within the cracks solidified their hypothesis. They concluded that the failure was initiated by hydrogen stress cracking, which resulted in crack formation. Subsequently, these cracks were subjected to corrosion, ultimately leading to the unexpected failure of the screws.

4 Failure Prevention

According to the conclusion reached by the research team, the failure primarily stemmed from the presence of hydrogen near the steel core of the screws. Therefore, one way to prevent crack formation would be to eliminate the presence of hydrogen. They attributed the source of hydrogen to the anodic and cathodic reactions occurring between the coating and the steel, which happened because the coating was exposed to the environment. In simpler terms, the paint applied to the coating did not completely cover it. Therefore, improving the quality of the paint and ensuring better coverage during the coating process could prevent such failures.

Additionally, environmental corrosion was identified as the secondary cause. To mitigate this, isolating the screws from the environment as much as possible would be a viable solution. For instance, in cases where the screws were exposed to rainfall, covering them to protect against rain exposure could help prevent future failures.

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Materials safety: article exercise

Failure analysis is an essential process for improving material selection and mechanical design, allowing us to learn from past failures to prevent future ones. In this report, we will analyze the failure presented in the article about M42 screws used in a power generator and explore the primary cause, ruling out alternate failure mechanisms, and providing recommendations to prevent similar failures in the future.

In the article the investigation involved a multi-faceted approach, including visual examination, material properties determination, fractographic and metallographic examination, and engineering analysis. With the information gathered from these investigation methods the primary cause of the failure was determined as follows.

From the background information we know that the surrounding conditions however were not ideal, the screw was exposed to rainwater and some residues that could lead to sulfuric acid formation. Upon visual examination, several key observations were made, indicating a failure linked to stress and corrosion simultaneously: The screws exhibited corrosion residues, the fractures had a fully brittle macroscopic appearance, crack nucleation occurred at the concordance radius between the screw body and head, and fracture surfaces showed advanced corrosion.

The material properties were determined with chemical analysis, hardness testing, and tension analysis were conducted to confirm the material specification of the screws. The material was found to be similar to AISI 4340 low alloy steel and mechanical properties aligned with class 8.8 screws resistance.

In the fractographic and metallographic examination the following observations were made: fractographic analysis could not provide detailed insights due to advanced fracture surface corrosion and mechanical damage. However, intergranular fracture with gaps between grains on preexisting cracks region was observed. Metallographic examination revealed a tempered martensitic microstructure and substantial degradation by intergranular cracks. The presence of corrosion products inside the cracks suggested a corrosion-related failure. The cadmium coating, predominantly composed of cadmium, was found to have localized corrosion points, particularly where the paint was faulty.

The primary cause of the failure of M42 screws can be attributed to hydrogen stress cracking predominantly, with concurrent corrosion processes like stress corrosion cracking. And the chain of action leading to failure is as follows:

1. The screws were exposed to unfavorable environmental conditions, including rainwater and residues that could lead to sulfuric acid solution formation.
2. Cadmium coating, applied to the screws for corrosion protection, was subject to localized corrosion due to paint damage, causing the steel beneath to be exposed to the environment.
3. The exposed steel underwent anodic dissolution due to the presence of cadmium coating as the anode and steel as the cathode in a galvanic corrosion setup.
4. Hydrogen was produced during this process, and it diffused into the steel, resulting in hydrogen stress cracking.
5. In addition to HSC, the open crack surfaces on loaded screws were exposed to a corrosive environment, causing corrosion products to form, and stress corrosion cracking may have occurred concurrently.

Alternative failure mechanisms:

1. Plastic deformation can be ruled out since there was no significant plastic deformation observed in the screws, and the failure was brittle.
2. Creep can be ruled out as the failure occurred suddenly, and creep is a time-dependent deformation phenomenon.
3. Creep can be ruled out as the failure occurred suddenly, and creep is a time-dependent deformation phenomenon.
4. Fatigue can be ruled out because the observed failure occurred suddenly and was not preceded by a period of cyclic loading or stress.
5. Environmentally assisted failure is the primary failure mechanism in this case, specifically hydrogen stress cracking and potentially stress corrosion cracking.

To prevent similar failures in the future, several actions can be taken:

1. Improved Coating: Enhance the quality and uniformity of the protective coating, such as cadmium, to ensure consistent corrosion resistance across the entire surface of the screw.
2. Regular Inspection: Implement a regular inspection and maintenance program to detect early signs of coating damage and corrosion. Replace screws with damaged coatings promptly.
3. Material Selection: Consider alternative materials with improved resistance to hydrogen embrittlement, such as stainless steel or nickel alloys, for critical applications.

4. Environment Control: Minimize exposure to unfavorable environmental conditions, especially those that can lead to the formation of corrosive solutions like sulfuric acid.
5. Monitoring: Implement continuous monitoring systems to detect changes in the environment and coating integrity, allowing for timely corrective actions.

In conclusion, the failure of M42 screws in the power generator was primarily due to hydrogen stress cracking (HSC) and potentially stress corrosion cracking (SCC) caused by localized corrosion of the protective cadmium coating and exposure to a corrosive environment. To prevent such failures, improvements in coating quality, regular inspections, alternative material selection, environmental control, and monitoring systems are recommended.

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Background

The article investigates failures in M42 bolts that were used in a power generator. The screws had fractured at the bottom of the screw head prematurely and unexpectedly. The generator was operated in conditions where the screws were exposed to rainwater and residues that might lead to formation of sulfuric acid solution.

Investigation methods

The fracture surfaces were at first examined visually, which revealed that the failure was linked to stress and corrosion. The screws had two means of protection against corrosion: electrochemical treatment (cadmium coating) and paint, which raises a question about how this was possible.

The material properties of the screws were verified with Rockwell hardness test, tensile test, and chemical analysis. The results indicated that everything conformed to class 8.8 screw manufactured according to ASTM F568M-07 standard.

Metallographic samples were prepared, and the microstructure of the steel examined under an optical microscope, confirming it to be tempered martensite, as expected and compatible with the standard.

A liquid penetrant inspection was conducted for intact screws recovered from the same generator to inspect for any cracking, but no cracks were found.

A tensile test was performed for an intact screw. A hole was drilled through the screw's head to decrease the cross-sectional area at the location at which the failed screws had cracked, ensuring the screw would fail at the same location. The resulting fracture surface had pre-existing, corroded cracks from which the failure progressed as ductile fracture during the tensile test, confirming that despite unsuccessful liquid penetrant test, intact screws had experienced cracking in the same location as the failed screws had fractured.

The fracture surface of the tensile test specimen was investigated under a backscattering electron microscope. The results show that pre-existing cracks had progressed as a brittle intergranular fracture. The cracks had residue buildup, which was analyzed using energy dispersive spectroscopy and confirmed to contain iron, nickel, cadmium, and oxygen. The results suggest both the steel and the cadmium coating had corroded. The corrosion build-up and the fact that the screw was no longer under high tensile load, as it would be when installed in the generator, explain why liquid penetrant testing was unsuccessful, as the cracks were closed and the penetrant fluid was not able to seep in.

Primary cause

The primary cause of the failure was determined to be hydrogen stress cracking (HSC). HSC progresses as an intergranular crack and is a typical cause for brittle fracturing of a normally ductile alloy under high load, which was the case for the screws.

HSC requires the presence of hydrogen, which had two potential sources in this case. The electrochemical coating process by which the cadmium coating was deposited on the screws generated hydrogen, which may then make its way into the base metal. However, as the cracking was very localized, this explanation is unlikely. If the hydrogen source was the coating process, the screws would have been susceptible to cracking across their whole surface area. The second option was the electrochemical corrosion of the cadmium coating, which was possible due to the corrosive environment the generator was operated in.

Normally the cadmium coating would corrode across a large surface area, which would mean that the coating would wear very slowly. However, as the screws were painted in addition to the cadmium coating, the coating was exposed to the corrosive environment in only very small areas where the paint was damaged. As the corrosion was localized to a small area, the coating corroded quickly, exposing the steel underneath. Hydrogen generated in the corrosion process of the coating then entered the steel, making it susceptible to HSC. After the cadmium coating had completely corroded away from the exposed area, the steel lost its protection against the corrosive environment, and HSC cracks started to form, ultimately leading to failure.

Alternate failure mechanisms

Plastic deformation due to overloading situation was ruled out as the damage was not consistent with the observed behavior in tensile tests (high elongation and necking).

The article considers stress corrosion cracking (SCC) as another possible mechanism for developing the cracks. SCC occurs in components that are subjected to a tensile load, which may be stable, and cracks were found in screws that were subjected to constant assembly load. However, if SCC was the cause, there would be corrosion at the crack tip, which was not the case in the investigated screws. Freshly progressed segments of the cracks were clean, and corrosion products appeared in areas that had been cracked for some time. Therefore, SCC was ruled out as a primary cause of the failure.

Prevention of similar failures

The failure was ultimately caused by two layers of corrosion protection (cadmium coating and paint) working against their intended purpose. If the screws were not painted, the coating would corrode faster overall, but over so much larger area that the service time before steel would get exposed could be significantly higher. On the other hand, if the non-coated screws were used, hydrogen would not be produced, and the screws would not become susceptible to HSC. Both options may produce too low service time, however.

The cracks nucleated at the concordance between screw body and head, where the paint was likely damaged upon tightening the screw. Rounding or chamfering the edge of the screw hole, and/or adding a washer to the assembly could be enough alleviate this issue without sacrifices in the level of corrosion protection. The downside of this option is that any other scratches in the paint could still lead to similar failures, as the root cause is not necessarily addressed.

A third option would be to use some other form of corrosion protection than a sacrificial coating. Barrier coatings could protect the screw without the risk of fast localized corrosion in areas where paint is damaged. Barrier coatings are typically some type of a polymer, which might not be suitable for coating a screw, especially the threads. Using stainless steel screws would provide a high level of corrosion resistance without the risk of a coating corroding through and exposing a base material to environment. Despite stainless steel screws being rather expensive, they are the most promising option for preventing further similar failures.

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1. Introduction

Stainless steel is widely used in the brewery industry because of its resistance to corrosion. In addition, well-chosen high-alloy steel allows for easier maintenance of hygienic conditions inside the tank [1]. An analysis of possible technological solutions indicates that 1.4301 steel is one of the most popular and cost-effective materials [2]. Corrosion problems in beer manufacturing are frequently reported. Both the beer manufacturing lines and the auxiliary infrastructure are at risk [3]. The data concerning problems with stainless steel resistance to corrosion depending on the surface treatment are also presented [4].

This paper presents the causes and a diagnosis of the corrosion types in a tank used to store warm water in a brewery house. Corrosion of the installation made of austenitic stainless steel in conditions of hot water containing chloride ions can be considered as a very common problem [5]. Detailed researches had been performed in order to solve this problem after it was found that corrosion phenomenon also concerns cold water installations in nuclear power plants [6,7]. It was recognised that it would be demanded to perform a very detailed selection of construction materials on the basis of chemical composition of water and its temperature [8,9]. Corrosion in hot water installations occurs mainly in the form of stress-corrosion cracking or pitting corrosion [10]. Nevertheless, the stress-corrosion cracking process can be initiated by the pitting corrosion, precisely by the increase in stresses in the pits' bottom [11,12]. Data available in scientific literature indicate that appearance of localised corrosion processes in hot water installations (static stresses) does not demand high concentration of chloride ions. Studies carried out by Turnbull et al. [11] point out the critical concentration of chloride ions at the level of 1.6 pp. [11].

The tank in question is made of 1.4301 (304 AISI) steel and has been in operation for 12 years. The investigated tank was made of 12 welded cylindrical elements of 1.4301 stainless steel of thickness from 2.5 mm at the upper part of the tank to 5 mm at the bottom part. The altitude of the tank was 18.6 m and cubic capacity was 200 m³. The water under hydrostatic pressure resulting from the actual level of water in conditions of 80 °C was stored in the examined tank. Walls of the bottom

cylindrical part of the tank were subjected to circumferential stresses, longitudinal stresses resulting from the interactions of hydrostatic pressure of water, and to the buckling forces resulting from the high altitude of the tank and the low thickness of the walls. In the case of stresses analysis, one should also consider changes of water temperature. Permissible stresses for 1.4301 stainless steel are decreasing by about 25% in the case of hot water storage as compared to the strength in conditions of cold water. It served as a storage tank for utility water used in beer mashing. The water temperature inside the tank was in a range of 70–80 °C. The water level in the tank was variable, and the water supply was connected to the upper part of the tank. The tank was not supervised during operation. As a result of its 11-year operation, a traverse crack occurred in the tank, causing a spill of utility water. Less than a year after that event, another crack appeared. Due to the extensive corrosion of other system components, there was the need to explain the causes of corrosion damage that threatens the safety of the manufacturing process.

2. Destruction of water storage tank

After a downtime and submitting the tank for inspection, the inner stainless steel surface was covered with dark brown deposit consisting mainly of the products of corrosion. The deposit was not very adhesive and could be easily removed with a paper towel. No changes in the steel below the deposit were found, so it can be concluded that the deposit was driven in by the water due to the corrosion in the water supply installation. The condition of the tank surface is shown in Fig. 1.

A corroded area was found in an approx. 1.5 m-wide cylindrical fragment of the tank (Fig. 2), located in the zone of mechanical impact of the pipeline outside the tank (See Fig. 3).

The locations of tank repairs correspond precisely to the zones of intense stress caused by the pipeline. This allows for associating the corrosion with the mechanical and corrosive impact. Corrosion attack visible in the form of branched lines and local centres is shown in Fig. 4.



Fig. 1. View of the inner surface of the container with partially removed deposit.

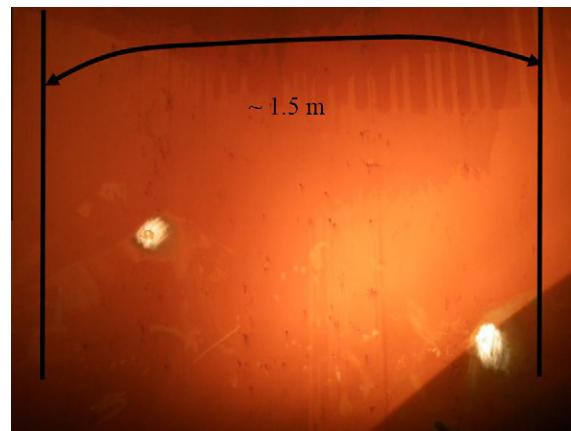


Fig. 2. A corroded part of the stainless steel tank.

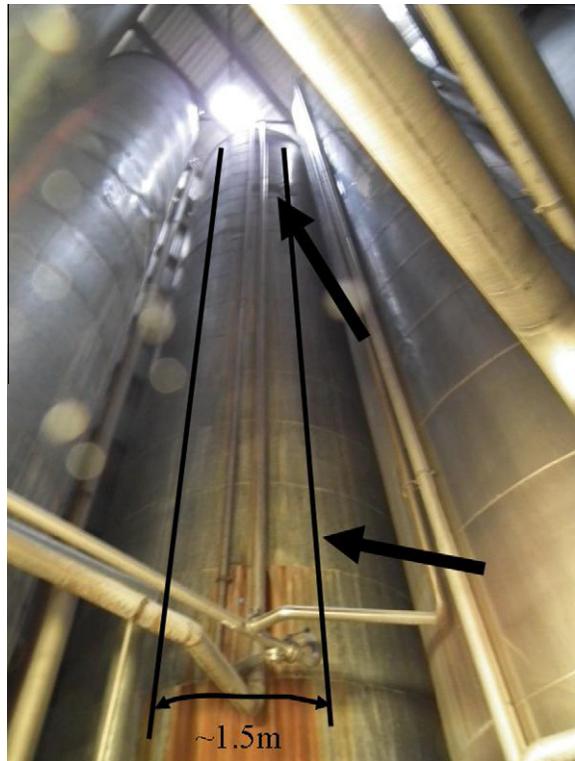


Fig. 3. Outside view of the tank with the corroded zone inside the tank marked.

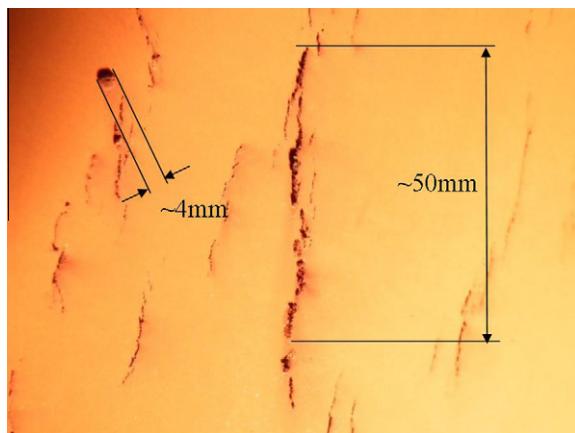


Fig. 4. Examples of corrosive attack of the cylindrical part of the tank.

After the products of corrosion were removed, clear corrosive attacks could be seen as cracks, pits and etchings (the result of deposit corrosion) (Fig. 5).

In some places, the cracks extended across the thickness of the tank. Fig. 6 shows the location of the total tank rupture (perforation).

Due to the presence of cracks visible on the tank surface, the stresses resulting from the interactions of hydrostatic pressure were calculated at the levels of 63% and 85% of the permissible tensile stresses for cold and hot water, respectively.

3. Experimental methods

Electrochemical investigations were carried out on 1.4301 stainless steel, from which the examined tank was constructed. The chemical composition of 1.4301 austenitic stainless steel is given in Table 1.



Fig. 5. Corrosive attacks in a stainless steel tank.



Fig. 6. Corrosion cracking of the mantle as the cause of perforation.

Table 1

Chemical composition of the austenitic stainless steel (AISI 304).

C	Si	Mn	P	S	Cr	Ni	Nb	Cu	Co	N
0.020	0.34	1.68	0.027	0.001	18.17	8.03	0.002	0.28	0.20	0.062

Cyclic polarisation tests were performed with the use of Gamry Instruments electrochemical measurement set-up. The tests were used for the determination of the susceptibility of the examined alloy to pitting corrosion and they were carried out in a three-electrode system. The scan rate of the potential was 0.5 mV/s. The examined electrode was the investigated stainless steel, the auxiliary electrode was made of platinum net and the reference electrode was a silver wire covered with silver chloride and placed directly in the solution used for tests. The susceptibility to pitting corrosion was determined in water before and after treatment. Comparative studies were also carried out for 1.4436 and 1.4462 stainless steels. The first type of steel is the potential material for the new tank, while the 1.4462 steel seems to be the most suitable material when considering the corrosion resistance and mechanical strength for applications involving water treatment.

Potential measurements of the inner surface of the tank were performed under field conditions. The value of the corrosion potential measured using a specially constructed reference electrode shows the quality of the passive layer covering the steel. The lower the value of the potential is, the greater the risk of corrosion. After it is emptied, the tank undergoes self-passivation due to the presence of air and moisture, which makes it possible to use the potential measurement to determine the locations, where the passivation conditions are hampered.

Additionally, SEM research of the examined steel surface was conducted. Steel specimens were taken from the places, where the corrosion products were detected on the tank. SEM images were obtained by using S-3400N Hitachi VP-SEM (with THERMO Scientific EDS unit attached). Results of this analysis are presented in Fig. 7.

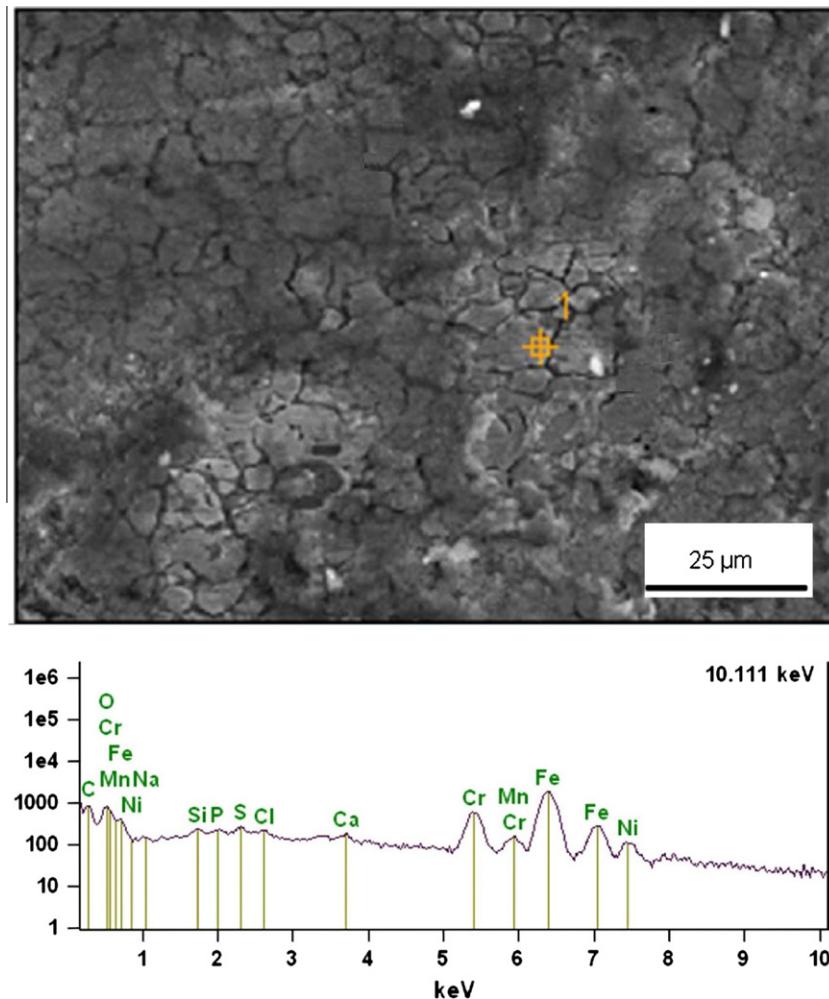


Fig. 7. SEM and EDS images of the corroded surface of the tank.

During the study of the buffer tanks, water samples were taken in order to conduct rudimentary analytical studies that would allow for the determination of the threat of corrosion associated with the influence of the water environment.

The water samples were taken from the following locations:

- Raw water taken before the water treatment system.
- Water taken from the water installation of the brewery house after treatment.
- Circulating water taken from a tank made of unalloyed constructional steel.

Aggressive ion contents in the raw water and in the treated water (after pH correction) are gathered in [Table 2](#).

In order to determine the corrosivity of water at the each stage of the beer production, corrosivity indexes for the investigated solutions were calculated in various temperatures. Temperature of raw water was about 7 °C. The Langelier index in such conditions was 0.08. As a result, the cold water could be determined as one of weak corrosivity with low tendency to form carbonate layers. Extrapolation of the calculated Langelier indexes to higher temperature improves their value.

Table 2

The results of chemical analyses of water samples.

	pH	Ca ²⁺	Mg ²⁺	Fe-total	SO ₄ ²⁻	Cl ⁻	O ₂	Cond.
Raw water	7.11	76	10	0.098	58	12.05	0.46	40.80
Treated water	5.86	61	8	0.04	45	66.72	0.55	32.50
Tank water	5.94	60	8	0.41	42	72.31	0.57	34.20

4. Experimental results and discussions

4.1. The study of the susceptibility of stainless steels to pitting corrosion in water prior to pH adjustment

The water in the brewery was subjected to the technology of changing its chemical composition in order to obtain the optimal parameters for beer-manufacturing purposes. It underwent water treatment and pH adjustment. Apart from the steel used to build the tank, the study also included the 1.4436 steel to be used as an alternative material for the future tank and the recommended 1.4462 steel with good mechanical properties and better corrosion resistance.

Tests of susceptibility to pitting corrosion in water before treatment were performed at 75 °C. This is a typical temperature of the water stored in the tank. [Fig. 8](#) shows example curves of the cyclic polarisation of stainless steels in water.

The studied steels remained in the passive state in these conditions. The increase of the current in the polarisation curves occurs at a potential of about 0.9 V and it is linked to the emission of gaseous oxygen on the surface of the working electrode. The polarisation curve after the reversal of polarisation clearly indicates the lack of pitting corrosion. Based on these results, it can be concluded that pre-treatment water is not aggressive. The probability of pitting corrosion occurring is nearing zero in all types of steel studied.

4.2. The study of the susceptibility of stainless steels to pitting corrosion in treated water

The cyclic polarisation measurements were also performed at 75 °C. [Fig. 9](#) presents the results of these tests.

In a pH-adjusted water, only duplex 1.4462 steel does not show any susceptibility to pitting corrosion. Microscopic evaluation of the steel surface has also not shown the presence of pitting. Therefore, it can be concluded that the steel is resistant to corrosion in treated water within the whole temperature range. 1.4301 and 1.4436 steel types are subject to pitting corrosion. The pitting nucleation potentials are:

- 1.4301 steel = + 0.30 V in Ag/AgCl.
- 1.4436 steel = + 0.45 V in Ag/AgCl.

The pitting nucleation potential for 1.4436 steel is higher than that for 1.4301 steel, which indicates a higher resistance to pitting corrosion and a lower probability of occurrence of this type of corrosion in treated water at 75 °C, but does not exclude the occurrence of such destruction.

After completing the electrochemical measurements, a photo of a 1.4301 steel sample was taken after studying it in water at 75 °C, which is shown in [Fig. 10](#).

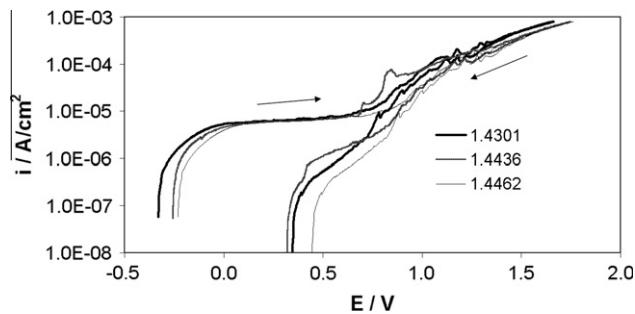


Fig. 8. Cyclic polarisation curves in raw water at a temperature of 75 °C for investigated steel.

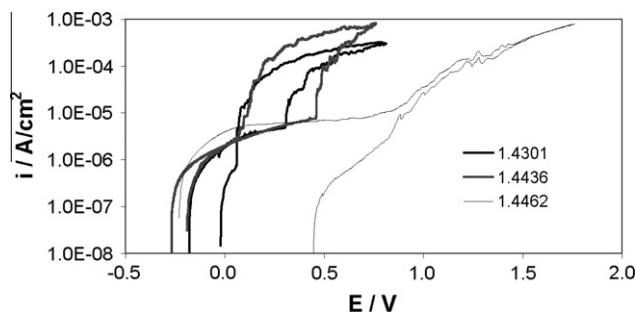


Fig. 9. Cyclic polarisation curves in treated water at a temperature of 75 °C.

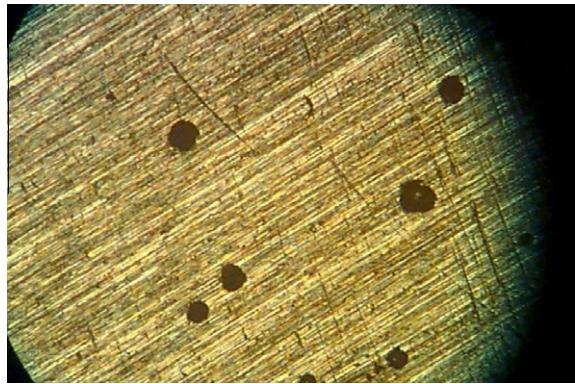


Fig. 10. The surface of a 1.4301 steel sample after cyclical polarisation test in water at 75 °C.

Fig. 11 shows a curve of pitting nucleation potentials as a function of the temperature of the treated water.

As shown in **Fig. 11**, the 1.4462 steel does not undergo pitting corrosion throughout the whole temperature range, and the probability of pitting corrosion occurring in 1.4436 steel is smaller than in 1.4301 steel.

4.3. Analysis of the chemical composition of water

After sampling the water before and after treatment process, basic analytical tests were made, that may be useful to determine the causes of corrosion problems in the installation. The results of the analyses are presented in [Table 2](#).

The chemical analyses of the treated water show a change in the pH value of approximately 7 to 5–5.5. This is caused by the dosing of concentrated hydrochloric acid. This results in a simultaneous increase in the Cl^- ion content in the water from 15–35 mg/dm³ to 55–100 mg/dm³. The pH change and the increase in the chloride ion content impact the corrosive aggressiveness of water. It should be noted that there is an additional increase of chloride ion content in the water that is circulating in the circuit ([Table 2](#)). The pH drop also accelerates corrosion in the installation, thus increasing by 10-fold the concentration of iron ions. The high chloride ion content increases the susceptibility of 1.4301 steel to pitting corrosion. Chloride ions weaken the passive film on stainless steel and cause the intensification of corrosion processes on non-alloyed steels. The stainless steels undergo pitting corrosion, while the low-alloy steels are subject to uneven general corrosion. The high level of chloride ions and the temperature of 75 °C create the conditions for initiation of both pitting and corrosive cracking.

4.4. The study of the potential of the inner part of the stack

The measurement results show that the tank is well-fitted. However, areas with a significantly lower potential (by as much as 0.3 V) can be found on the surface. These are probably places, where pitting corrosion develops. The potential studies have shown that the passive condition of the tank is much better in the upper parts. The potential measurement results are summarised in [Table 3](#).

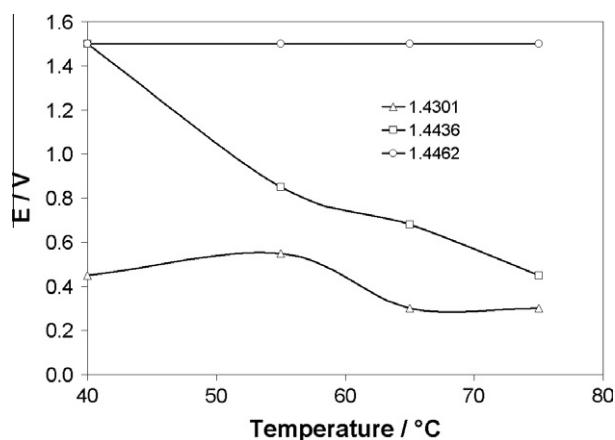


Fig. 11. Changes of pitting nucleation potentials vs. the temperature of the treated water.

Table 3The results of potential measurements of the tank walls a Zn/ZnSO₄ electrode.

Location of measurement	E/V
The area around the welded joint between the first and second sheet in the cylindrical part of the tank	1.340, 1.330, 1.281, 1.256, 1.237, 1.244, 1.329
The sheet of the second cylindrical part of the tank	1.257, 1.190, 1.197, 1.181, 1.175, 1.179
Polished area on the sheet of the first cylindrical part of the tank	1.215, 1.217, 1.237
The sheet of the first cylindrical part of the tank	1.200, 1.228, 1.218, 1.206, 1.171
The area around the weld joining the bottom with the cylindrical part of the tank	1.167, 1.202, 0.865 , 1.184, 1.279, 1.191, 1.193
The area around the weld of the two bottom parts	0.979 , 1.278, 1.015, 1.217, 1.057
Tank bottom	1.198, 1.171, 1.030, 1.050, 0.985

Bold values indicate places where passive layer is especially weak.

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Materials safety: article exercise

You are working as a materials expert in your organization and your responsibility is to guarantee safe and efficient use of materials in your facility. One day, a failure in similar facility is brought to your attention, and you need to investigate the possible implications this failure has for your facility. Your job is to interpret and analyze the given failure report and to write a report, which will allow others in your organization to understand the key developments and causes leading to the failure and the necessary actions for prevention of such failures.

Read the given article and analyze the failure. Describe, how the deformation and failure mechanisms presented during the course are reflected in the case and establish the chain of actions leading to the failure. The report should work as an introductory material to your team; it should not be very long but it should enable other team members to understand the key features of the failure without reading the failure report itself.

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Prepare your response by editing this word document and export it as PDF. The file name identifies you and the article. Do not change the file name (other than the extension to pdf). E-mail the pdf to "materials.safety@iikka.fi".

Name: Nguyen Xuan Binh
Student ID: 887799

Materials Safety analysis report on corrosion of brewery tankers

Problem Overview of the report:

The report discusses a problem faced by the brewery industry concerning corrosion in buffer tanks. Stainless steel is a popular choice in breweries for the water tanks for its corrosion resistance, but it has been observed to experience corrosion issues after 12 years in operation. The report outlines a specific case where the tank used to store warm water in a brewery exhibited signs of corrosion. Through different analyses, the report seeks to diagnose the types of corrosion and their underlying causes, emphasizing the role of possible factors in corrosion such as water treatment, pH adjustment, and the presence of chloride ions.

A. Description of investigation methods applied

- What means of investigation were used in the failure analysis?

The author tries to determine whether the threat of corrosion is associated with the presence of the water environment surrounding the tanks. From the report, the means of investigation for the failure analysis included:

1. Observations on the cracks: After removing the corrosion deposits with a paper, the tank reveals the cracks, pits, and etchings, possibly caused by the chloride water.
2. Chemical Composition Analysis: The author provides the chemical composition of the 1.4301 austenitic stainless steel, which can account for its specific corrosion impact.
3. Stress calculations: due to the presence of visible cracks on the tank surface, the stresses resulting from hydrostatic pressure were calculated, which were reported to be within permissible limits of hot and cold water.
4. Electrochemical Investigations: these were carried out on the 1.4301 steel, which was the material from which the examined tank was constructed. This is the main methodology in the report to study the chemical properties of the contained water.

- What computational methods were used?

The author lists a couple of computation methods as follows:

1. Cyclic polarization tests on three-electrode system with the use of Gamry Instruments electrochemical measurement to determine the susceptibility of the alloy to pitting corrosion.
2. Langelier corrosivity indexes were calculated in different temperatures to determine the water corrosivity at various stages
3. Scanning electron microscope (SEM) research was conducted on the steel surface. This studied material was extracted from where the corosions occurred on the tank.

- What material or results were obtained?

The author studied 3 different stainless steels: 1.4301 (current in use), 1.4462 (candidate) and 1.4436 (candidate). After the analyses, the author obtained the results based on 4 criteria

1. Susceptibility of 3 candidate stainless steels to pitting corrosion by water at 75 °C prior to pH adjustment: the author concludes pre-treatment water is not aggressive. The probability of pitting corrosion is extremely low for all 3 steels.
2. Susceptibility of 3 stainless steels to pitting corrosion by PH-treated water at 75 °C: only 1.4462 steel is resistant against pitting corrosion, while the others are susceptible.
3. Chemical composition of water: After treatment, the water's pH value decreases from 7 to 5–5.5 level, which increases the chloride ions concentration, making the water more corrosive. The studied steels become more susceptible to pitting and corrosive cracking.
4. Electrical voltage of the stack's inner part: areas with lower potential voltage are more likely to be impacted by pitting corrosion, such as the upper parts of the tanks. It is noted that 1.4301 steel potential is constant with regards to varying temperatures.

The author believed that the tanks should be built with two new austenitic steels instead of the current austenitic steel. Even though these new materials are more costly, it is worthwhile in the long run as they are highly durable against corosions.

B. The primary cause of the failure and description of the failure mechanism

- What is the primary cause of the failure (also provide reasoning)?

The primary cause of the failure in the study is most likely to be associated with various types of corrosive attacks on the tank. These corrosive attacks, including cracks, pits, and etchings, are attributed to deposit corrosion. The deposit corrosion was driven by the water due to corrosion in the water supply installation. Furthermore, the locations of tank repairs corresponded precisely to the zones of intense stress caused by the pipeline, suggesting a coupled mechanical and corrosive impact as contributing factors to the failure.

The paper did state a line as follows:

“The locations of tank repairs correspond precisely to the zones of intense stress caused by the pipeline. This allows for associating the corrosion with the mechanical and corrosive impact.”

This indicates that the primary cause of the tank's failure was a combination of mechanical stress (such as circumferential, longitudinal, and buckling stress) upon the pipeline wall and corrosion by the water supply installation.

- What's the chain of action that led to the failure?

The chronological order of the tank failure is as follows:

1. Deposit Formation: The inner stainless-steel surface of the tank was covered with a dark brown deposit, primarily composed of corrosion products. This deposit was driven into the tank by the water due to corrosion in the water supply installation.
2. Mechanical Impact: An area of the tank, approximately 1.5 m wide, was in the zone of mechanical impact from an external pipeline. Additionally, hydrostatic pressure also causes great pressure on the bottom of the tanks.
3. Coupled effect: The locations of tank repairs matched the zones of intense stress, suggesting that the corrosion was exacerbated by both mechanical and corrosive impacts.

4. Visible corrosive marks: After removing the corrosion deposits, clear cracks and pits were observed, such as a large crack of 50 mm and a small one of 4 mm. Water can leak out from the tank through these cracks. This is the final stage of the tank's failure.

C. Ruling out alternate failure mechanisms

- Can plastic deformation be ruled out? If yes, explain how.

Plastic deformation occurs when the material deforms pass its yielding strength. This can be ruled out as the stresses resulting from hydrostatic pressure were calculated and were found to be within permissible limits for both cold and hot water, suggesting that the stresses were not sufficient to cause plastic deformation.

- Can creep be ruled out? If yes, explain how.

Creep is the slow deformation while the material is subject to persistent mechanical stresses. It can result in failure even if the applied stress is below the yield strength of the material due to long term load exposure. This failure mechanism in my opinion should be seriously considered. The brewery tank is continuously subjected to internal pressures due to the stored warm water. Over time, even if the stresses induced by this pressure are below the yield strength of the stainless steel, the stainless steel can start to be affected by creep. Additionally, creep is more serious at higher temperatures if the warm water in the tank is maintained isothermally. Also, the paper provides that the tank has been in use for 12 years, which fits the time frame of creep.

- Can brittle fracture be ruled out? If yes, explain how.

Brittle fracture can be ruled out since stainless steel is typically ductile at room temperature to warm temperature. Brittle fractures are more likely to occur under high strain rates, such as large impact loadings. In the case of the brewery tank, the operational conditions involve relatively slow and steady internal pressures, leading to low strain rates. If anything, this load could have caused ductile deformation before the brittle fracture.

- Can fatigue be ruled out? If yes, explain how.

Fatigue can be ruled out because there is no description of cyclic loading against the tank wall. The mechanical stress on the wall is mostly constant such as hydrostatic pressure.

- Can environmentally assisted failure be ruled out? If yes, explain how.

Environmentally assisted failure, specifically in the form of corrosion, was the primary concern in the report. The corrosive attacks were driven by the water due to corrosion in the water supply installation. Therefore, this failure should be the top primary concern of the failure. Besides corrosion, environmentally assisted failure also includes hydrogen embrittlement. However, this is not the case here as the report finds that the chloride ions chiefly make the water corrosive.

The paper explicitly states as follows:

"No changes in the steel below the deposit were found, so it can be concluded that the deposit was driven in by the water due to the corrosion in the water supply installation"

D. Recommendations to prevent similar failures in the future

- How should the design, material, use, etc. be developed to avoid similar failures in the future? Provide several alternatives and indicate the most promising.

To avoid similar failures in the future, several design, material, and usage recommendations can be inferred from the paper

1. Material Selection: The paper suggests considering alternative stainless-steel materials. Specifically, the study included 1.4436 steel as a potential alternative material for future tanks. Additionally, 1.4462 steel was recommended due to its good mechanical properties and superior corrosion resistance.
2. Water Treatment: Ensure that the water used in the brewery undergoes appropriate PH treatment to minimize its corrosive aggressiveness. The study emphasizes the chemical composition of water and its temperature to prove its corrosive property.
3. Stress Analysis: Conduct thorough stress analyses, especially in areas where mechanical impacts are expected, such as near pipelines or the bottom of the tank. This can help in identifying potential weak points and reinforcing them as needed.
4. Water tank design: The water tank is high over 18 meters and has a large diameter with over 200 cubic meter volume of water, while the wall is very thin. This is quite dangerous as the load is very large against the not so reinforced tank wall. A good idea is to increase the wall thickness or reduce the height of the tank.
5. Environmental Considerations: Given that the environmentally assisted fracture played a significant role in the observed corrosion, it's crucial to monitor environmental factors, such as chloride ion content in the water, which can intensify corrosion processes.

The most promising methods are the first and second methods, since they are straightforward, easy to implement and relatively low-priced solutions.

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Materials safety: article exercise

Introduction

Out of the inspection from the tank, it was found that the tank was partly corroded from the inside, which had caused the cracks and leaks to the tank located in our brewery. We started the inspection after the cracks and leaks were found from the tank, in addition to the information which showed that the conditions in our tank were favorable for corrosion.

What had caused the failure?

It was found that the corrosion had occurred only partly inside the tank, but the deposit cause by the corrosion had spread all over it, even though it could be swept away by paper. The area which had corroded was around 1,5 meters wide section through the tank in its axial direction. In the corroded area, but outside of the tank, a pipeline was connected to the tank, which had caused more stresses to that area. Also, circumferential stresses, buckling forces and longitudinal stresses were found to be present in the tank walls.

It was able to be concluded that the corrosion was the result of both the corroding chemical reaction and the mechanical stresses. The corrosion was able to be seen as cracks, pits and etchings in the corroded area. Some of the cracks were also gone through the tank wall.

Another factors, which were partly causing the failure were the chloride-ion concentration of the water in the tank, in addition to the high water temperature, which was between 70 and 80°C. The need for chloride ions is smaller in hot water to cause corrosion, which had also increased the likeliness for the failure. Also, the stress corrosion has been found to be more likely in hot water environment, which can be seen also from the investigation of our brewery's tank.

Investigation methods

To find out the tendency of the tank material to the pitting corrosion, we conducted cyclic polarization tests. The pits have been found to cause stress-corrosion cracks, through which it was more reasonable to conduct the tests. The tests were done also for 1.4436 and 1.4462 stainless steels, out of which the 1.4436 was found to be potential material for the new tank.



To find out the quality of the passive layer inside the tank, potential measurements were done. Low potential was meaning higher risk for corrosion, and it also helped to see, where the passive layer was poor. The results showed that the lowest potentials located in the bottom of the tank and weld joints near the bottom.

Steel specimens taken from the corroded areas were investigated by using scanning electron microscope, to see the composition of the corroded areas better.

In addition, to know better the effect of water environment, water samples were taken from raw water before treatment, water installation of the brewery and circulation water from tank which was made of unalloyed constructional steel.

572266

Materials safety: article exercise

You are working as a materials expert in your organization and your responsibility is to guarantee safe and efficient use of materials in your facility. One day, a failure in similar facility is brought to your attention, and you need to investigate the possible implications this failure has for your facility. Your job is to interpret and analyze the given failure report and to write a report, which will allow others in your organization to understand the key developments and causes leading to the failure and the necessary actions for prevention of such failures.

Read the given article and analyze the failure. Describe, how the deformation and failure mechanisms presented during the course are reflected in the case and establish the chain of actions leading to the failure. The report should work as an introductory material to your team; it should not be very long but it should enable other team members to understand the key features of the failure without reading the failure report itself.

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If the author of the failure analysis has, in your view, neglected to address some aspects of the failure, you may indicate this in your report and suggest tests or actions that should have been done to clarify the issue.

Prepare your response by editing this word document and export it as PDF. The file name identifies you and the article. Do not change the file name (other than the extension to pdf). E-mail the pdf to "materials.safety@iikka.fi".

You may use the question list below to guide you in your analysis:

- A. Description of investigation methods applied
 - What means of investigation were used in the failure analysis?
 - What computational methods were used?
 - What material or results were obtained?
- B. The primary cause of the failure and description of the failure mechanism
 - What is the primary cause of the failure (also provide reasoning)?
 - What's the chain of action that led to the failure?
- C. Ruling out alternate failure mechanisms
 - Can plastic deformation be ruled out? If yes, explain how.
 - Can creep be ruled out? If yes, explain how.
 - Can brittle fracture be ruled out? If yes, explain how.
 - Can fatigue be ruled out? If yes, explain how.
 - Can environmentally assisted failure be ruled out? If yes, explain how.
- D. Recommendations to prevent similar failures in the future
 - How should the design, material, use, etc. be developed to avoid similar failures in the future? Provide several alternatives and indicate most promising.

1 Introduction

This report reviews the failure analysis of a facility water tank failure. Water tank is used to store warm water. The stored hot water contains chloride ions that are very corrosive.

Water tank here is made of 1.4301 (AISI 304) stainless-steel that houses $200\ m^3$ of water. The water tank is 18.6 meters long and made of welded cylindrical stainless-steel plates that are 2.5 mm thick on top and 5.0 mm thick in lower proportion of the water tank. In the facility, the water tank has been used for 12 years.

Due to its large size and application, the water tank is vulnerable for various stresses and failure mechanisms. From mechanical point of view, the $200\ m^3$ of water causes hydrostatic pressure within the tank that causes stresses within the water tank. Failure of exceeding hydrostatic pressure would result e.g., fracture in stainless-steel plate. Hydrostatic pressure with hot and cold water were calculated to cause tensile stresses of 63 to 85 % of permissible levels. Water tank does also get emptied that alters the water tank for failure due to fatigue i.e., mechanical failure due to alternating stresses.

From thermal and chemical perspectives, author of failure analysis does suggest considering thermal fatigue in failure analyses. In the water tank failure, water temperature did alter from 80 °C to cold water. Chemical composition of water created good environment for corrosion as the water sample from the tank showed high concentration of chlorine ions due to presence of hydrochloric acid.

2 Investigation methods

The author conducted a study to investigate the water tank failure. Intuitively, the author began study by inspecting the water tank that had clear signs of corrosion inside and outside the tank. Corrosion inside the tank was clearly at the water level when the tank was full. The author inspected the outer shell of the tank and found large number of wide cracks that were around 50 mm long.

From experimental studies, the author took three samples of the circulating water systems and found out that the water has corrosive elements. Both treated water and tank water contained chlorine ions that indicate the presence of hydrochloric acid. Elevated temperature and presence of chlorine ions make the stainless steel vulnerable for pitting corrosion and corrosive cracking.

The author also performed cyclic polarization test to measure the 1.4301 stainless-steel vulnerability for corrosion rate. Other stainless-steel materials, such as 1.4436 and 1.4462 were also studied. 1.4301 is susceptible for stress corrosion cracking and has low resistance pitting corrosion.

3 Failure analysis

The failure mechanism seems to be tied to the corrosive properties of the water and vulnerability to corrosion of the water tank. As pointed out, the water stored in the water tank was aggressively corrosive that caused pitting corrosion. The 1.4301 stainless steel used here is susceptible for pitting corrosion and has a low resistance for stress corrosion cracking [Granta EduPack, 2023].

In the presence of high hydrostatic stresses, the failure has most likely happened due to three phases. First, the crack has formed due to pitting corrosion on the shell of the tank that is the primary cause of failure. Cracks have grown due to stress corrosion cracking with presence of hydrostatic pressure. Usually failure due to corrosion cracking is unexpected. When the crack has grown large enough and weakened the tank shell, hydrostatic pressure can penetrate the wall.

As always, there are possible other alternative failure mechanism that could have cause the failure. The considered failure mechanisms are listed below.

- Plastic deformation can be ruled out as the primary failure mechanism. If we look at the figure 6 in the authors analysis, the crack that caused the tank to fail has clear signs of corrosion. Plastic deformation could have happened if the stresses caused by the hydrostatic pressure would have exceeded the yield strength of material. However, the safety factor was over 1.1 with both hot and cold water. The failed crack in figure 6 shows small sign of sheet metal bent around the crack. Welded areas could be a week spot.
- Creep can be ruled out. Temperature gradient is relatively small and 80 degrees maximum. There were no direct signs of plastic deformation of the water tank.
- For brittle fracture, the stainless steel tank would have have to harden somehow during operation. 1.4301 cannot be heat treated. Other method is an error in manufacturing the water tank.
- Fatigue is highly unlikely to have happened. This is because 1.4301 stainless-steel has a relatively high fatigue strength [Granta EduPack, 2023] with low number of cycles. The tank has been used for 12 years, but the number fills and thermal cycles are most likely not in the range of 100 000s.
- Environmentally assisted failures can be ruled out. The water tank is located inside a facility with controlled climate.

The water tank should be constructed with more corrosion resistant material such as 1.4462 stainless-steel as this is rated as non-susceptible for pitting corrosion and high resistance for stress corrosion cracking. Strength of 1.4462 is lower than 1.4301 but this can be compensated with thicker wall design.

100542646

Materials safety: article exercise 1

Electrochemical investigations were carried out on 1.4301 stainless steel, which was used for the material construction (tank). Other steels (possible candidates for new tank material) 1.4436 and 1.4462 were also tested with cyclic polarization tests. Cyclic polarization test is often used to study corrosion on surface. In cyclic polarization test the voltage is swept through the range, returning to the starting potential. The surface is likely to be changed by the reactions during the scan (corrosion), so the return voltage may not "match" the data from the forward sweep. Higher potential, more corrosion resistance material.

Analytical tests were performed for the water. Tests were done for raw water, water taken from the installation of brewery house after treatment and circulating water from the tank. The Langelier (corrosivity) index was calculated for all these waters. The water was heated to 75 degrees during the tests.

Results obtained were that steel 1.4301 is well suitable for raw untreated or low Langelier index water. For more corrosive water ($\text{pH} < 6$, high chloride ion content), the 1.4301 or 1.4436 steel is not suitable due to increase in susceptibility of pitting corrosion in the hole temperature range. It weakens the passive film on stainless steel. Best suitable steel found was 1.4462.

Primary cause of the failure was a material selection fault. The tank was not designed for such an aggressive water type. Steel 1.4301 was not able to withstand the constant stresses and was prone to corrosion. Plastic deformation can be ruled out, the 1.4301 steel did not achieve its total yield strength. Creep and brittle fatigue may have had some effect in chain action which led to the failure. The steel was prone to creep and brittle fatigue due to constant high stresses and maybe leftover internal stresses from welding. There were no signs of external damage to the tank.

To prevent similar failures in the future, corrosion should be taken more seriously into account, and especially investigate the environment the tank is installed.

101847162

Report

Floris Belle

Corrosion problems in beer manufacturing are often described. For instance, a water tank in the brewery, made from 1.4301 stainless steel. This tank which is being used to store warm water, faces some corrosion problems. The corrosion problems are being caused, because a non-suitable steel-alloy has been used to fabricate the storage tank. The storage tank can namely not withstand changes in pH-level, which are necessary in order to obtain the best parameters for brewing beer. After conducting a cyclic polarisation test in order to determine the susceptibility of various steel-alloys with regarding to pitting corrosion, it appears that a tank made of 1.4301 stainless steel, could namely not be corrosion-proof if the pH-level in the tank changed from 7 to 5.5. This change caused an increase of chloride ions in the water, due to a more acid environment in the tank. A too critical concentration of chloride ions increases the chance of weaken the passive film on the surface of the stainless steel, which normally would protect the metal against corrosion. If the metal does not contain a oxide layer anymore, pitting corrosion could start to show up. A storage tank made of 1.4301 stainless steel therefore experienced pitting corrosion as the culprit. There also appears to be some cracks present, which could be initiated by the additional stress in the pits' bottom. Temperature differences also occurred, which could cause extra material stresses, due to the expansion and shrinking of the tank. Moreover, hydrostatic pressures could play a role. Since the tank was not supervised during operation, the entire corrosion process had time to develop with all its consequences.

However, in contradictory to the 1.4301 stainless steel-alloy, the 1.4462 alloy performed well in pH-adjusted water and did not show any susceptibility to pitting corrosion and would therefore be a better choice of material than the 1.4301 steel-alloy. Also, the tank should be monitored during operation mode.



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Read the given article and analyze the failure. Describe, how the deformation and failure mechanisms presented during the course are reflected in the case and establish the chain of actions leading to the failure. The report should work as an introductory material to your team; it should not be very long but it should enable other team members to understand the key features of the failure without reading the failure report itself.

In addition to establishing the primary cause of the failure, show why alternate failure mechanisms can be ruled out. Some failure mechanisms have not been discussed in the course yet. Conduct the analysis using your present knowledge on the subject.

If the author of the failure analysis has, in your view, neglected to address some aspects of the failure, you may indicate this in your report and suggest tests or actions that should have been done to clarify the issue.

Prepare your response by editing this word document and export it as PDF. The file name identifies you and the article. Do not change the file name (other than the extension to pdf). E-mail the pdf to "materials.safety@iikka.fi".

You may use the question list below to guide you in your analysis:

A. Description of investigation methods applied

- What means of investigation were used in the failure analysis?
- What computational methods were used?
- What material or results were obtained?

B. The primary cause of the failure and description of the failure mechanism

- What is the primary cause of the failure (also provide reasoning)?
- What's the chain of action that led to the failure?

C. Ruling out alternate failure mechanisms

- Can plastic deformation be ruled out? If yes, explain how.
- Can creep be ruled out? If yes, explain how.
- Can brittle fracture be ruled out? If yes, explain how.
- Can fatigue be ruled out? If yes, explain how.
- Can environmentally assisted failure be ruled out? If yes, explain how.

D. Recommendations to prevent similar failures in the future

- How should the design, material, use, etc. be develop to avoid similar failures in the future? Provide several alternatives and indicate most promising.

100488467

Materials safety: article exercise

Investigation of the tank was carried out by firstly removing it from operation and submitting it for inspection. By visual inspection of the tank, deposits covering the inner surface were found, as well as a 1.5m wide corroded area. Removing the products of corrosion visualized corrosive attacks such as cracks, pits and etching. Scanning electron microscopy (SEM) and energy dispersive X-ray spectroscopy (EDS) was conducted on the examined material surface. Specimens for investigation were taken from areas with corrosive products.

To examine susceptibility to pitting corrosion of the material used in the tank, 1.4031 (304 AISI) austenitic stainless steel, cyclic polarization tests were carried out using electrochemical measurements. Susceptibility for pitting corrosion was examined in the water before and after treatment. This was also done for 1.4436 and 1.4462 stainless steels as a comparative study. Analytical studies of water samples were done to determine possible threat of corrosion due to the water environment. Samples were taken from raw water, water from the installation after treatment, and circulating water from a tank made of unalloyed constructional steel.

Primary cause of failure was related to high level of chloride ions and a temperature of 75°C. These conditions create circumstances that enhance susceptibility for both pitting and corrosive cracking. High level of chloride ions was found in the treated water, where dosing of concentrated hydrochloric acid caused a drop in pH level. This indicates that the failure is of environmentally assisted type, namely stress corrosion cracking (SCC). SCC is a failure mechanism where the following three conditions are present; susceptible material, an aggressive corrosive environment, and a sustained tensile stress.

From the analysis it was concluded that pre-treatment water would not increase probability of pitting corrosion. From the analysis, it was found that only duplex 1.4462 steel showed no susceptibility to pitting corrosion, and it was concluded that this material is resistant to corrosion in treated water within the whole temperature range. To avoid similar problems in the future, corrosion resistance and mechanical strength of the material should be considered for applications where water treatment is necessary.

427214

Article review: Brewery tank failure

Description of applied investigation methods

Visual observations showed brown corrosion deposits on the tank surface. There were cracks, pits and etchings that showed corrosion damage. Locally, the cracks went through whole wall thickness. Damage was found close to the parallel piping to the tank.

Electrochemical investigations for material composition. The tank was confirmed to be 1.40301 austenitic stainless steel.

Cyclic polarization tests were used to compare the used material to other potential replacements. The test determines a material's resistance to corrosion.

Scanning Electron Microscopy was used to examine material surface and damage.

The primary cause of the failure and description of the failure

The failure can be explained with corrosion. There were clear signs, such as clear corrosion deposits, cracks and pitting. The presence of chlorine ions in the water weakened the passive film on the surface of the stainless steel, enabling corrosion to occur. The cracking also implies that the stresses enabled SCC to occur.

Ruling out alternate failure mechanisms

Creep can be ruled out since the temperatures are relatively low.

Fatigue can be ruled out, since the loading cycle in the tank can be assumed low both in number of cycles and stress amplitudes.

Plastic deformation can be ruled out, since there were no large loads imposed on the tank.

Brittle failure is also not relevant due to temperature and load ranges.

Recommendations to prevent similar failures in the future

A better material was found, so that should be switched when making new tanks.

The corrosion occurred at the bottom, where other pipes were running parallel to the tank. Loads from those pipes were used as an explanation with hydrostatic pressure. Thus, by lifting these pipes higher would prevent described loads.

For existing tanks, NDT testing during operation should be introduced in the known areas of interest. Eddy-current testing for example could be used to find cracks and initiating pits. If possible, empty tanks could also be examined from inside during maintenance.

612524

Materials safety: article exercise

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Prepare your response by editing this word document and export it as PDF. The file name identifies you and the article. Do not change the file name (other than the extension to pdf). E-mail the pdf to "materials.safety@iikka.fi".

Failure report in question studies brewery water tank decommissioned due to corrosion cracking. Report mostly studies corrosion properties of 1.4301 austenitic stainless steel when subjected to chloridic and otherwise corrosive water in elevated temperatures. While the expected critical failure mode, rupture of the tank, is clearly mechanical in nature, no details of analysis are to be found.

Report includes an extensive study into corrosion of the stainless steel in conditions replicated from the actual processes. Tank was not the only corroded part, but the whole system is said to have been highly corroded. 11 years is a long time for any system and it is not clear how the systems have been inspected, even though accumulation of corrosion product could also present a problem with hygiene in a foodstuff production facility.

Corrosion tests were conducted with cyclic polarisation testing. Scanning electron microscopy and energy dispersive spectroscopy tests were also done to determine the accurate chemical composition of the steel. No abnormalities in chemical composition or microstructure were reported.

Tests indicated pitting corrosion should not occur in the conditions inherent to the process. However, lower potentials were measured in parts of the tank and hypothesized to be starting points of corrosion. The tendency of chloride ions to weaken the passivated layer on the surface of stainless steel was also noted.

Report comes to the conclusion that on areas of lower potential, under the effect of passivated-layer-weakening chloride ions, the mildly corrosive water allowed pits to form. These acted as stress risers for the hydrostatic pressure-induced stress, and in a corrosive environment, a fracture will propagate below the critical stress intensity factor. This is believed to have led to the fracture growing through the tank wall, resulting in a leak.

No load history or spectrum is specified to warrant any kind of fatigue analysis. In fact, nothing more than the presence of hydrostatic pressure is stated. It is stated that under hydrostatic pressure, tensile stresses reached 63-85% of "allowed". The type of limit state is not specified. How this stress was calculated, is not specified. Wording "allowed tensile stress" does not give confidence that a fracture mechanics analysis was conducted, even though a singular fracture has been identified and corrosion cracking is suspected. Subcritical stress intensity factors, i.e., the critical stress intensities for crack growth in a corrosive environment have been determined experimentally and could be utilized.

The hydrostatic load calculation should be documented, alongside possible variation to determine possible fatigue. It is likely no data is available of the load history, but the brewery likely could provide (with consulting from the analyst) a typical spectrum from a similar process. Possible vibration from pumps, thermal cycling or for example water hammer effects should also be taken into account.

101843742

Materials safety: article exercise

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You may use the question list below to guide you in your analysis:

A. Description of investigation methods applied

- **What means of investigation were used in the failure analysis?**

Cyclic polarization tests performed with the use of a Gamry Instruments electrochemical measurement set.

Water sampling at different points of the process.

Chemical analysis of water samples to determine their composition.

Measurement of the corrosion potential on the internal surface of the tank.

- **What computational methods were used?**

Computational methods are not specifically mentioned in the text, so no information on their use is provided.

- **What material or results were obtained?**

Determination of pitting corrosion susceptibility of different types of stainless steel in untreated and treated water conditions.

Analysis of the chemical composition of water at different stages of the brewing process.

Evaluation of the quality of the passive layer covering the stainless steel in the tank.

B. The primary cause of the failure and description of the failure mechanism

- **What is the primary cause of the failure (also provide reasoning)?**

The primary cause of failure is corrosion, specifically pitting corrosion. Pitting corrosion is a process in which small areas of the surface of a material, in this case stainless steel, corrode in a localized manner, forming holes or pits.

- **What's the chain of action that led to the failure?**

The failure mechanism involves several factors:

1. The water stored in the tank was in the 70-80°C temperature range.
2. The water experienced a change in its chemical composition due to the treatment process and pH adjustment, which included the addition of concentrated hydrochloric acid.
3. This change in water composition resulted in an increase in the chloride ion content in the water, which increased the corrosive aggressiveness of the water.
4. The high water temperature and the high chloride ion content weakened the passive layer that protects the stainless steel against corrosion.
5. As a result, pitting corrosion started on the stainless steel of the tank.
6. The hydrostatic pressure of the water stored in the tank, combined with the mechanical stresses due to the tank construction and contact with the piping system, aggravated the corrosion and led to the formation of cracks and holes in the stainless steel.

In summary, the main cause of failure was pitting corrosion induced by the combination of high water temperature, high chloride ion content and mechanical stresses in the tank. This process weakened the structural integrity of the tank and eventually led to tank failure.

C. Ruling out alternate failure mechanisms

- **Can plastic deformation be ruled out? If yes, explain how.**

Yes, plastic deformation can be ruled out as the primary cause of failure. The reason is that there is no indication in the text that the stainless steel experienced significant plastic deformation before the failure. Additionally, visible plastic deformations on the tank's surface or in the analyzed steel samples are not mentioned.

- **Can creep be ruled out? If yes, explain how.**

Yes, creep can be ruled out as the primary cause of failure. Creep is a long-term deformation process under constant or cyclic loads at high temperatures. In this case, although the water temperature in the tank was high (70-80°C), there is no mention of the stainless steel being under long-term constant or cyclic loads that could have caused creep. Furthermore, typical symptoms of creep, such as gradual elongation over time, are not mentioned in the text.

- **Can brittle fracture be ruled out? If yes, explain how.**

Yes, brittle fracture can be ruled out as the primary cause of failure. Brittle fracture is characterized by sudden and brittle material failure without significant deformation. In this case, the text describes localized corrosion and the formation of cracks and holes in the stainless steel, indicating a failure process consistent with corrosion and pitting corrosion, rather than brittle fracture.

- **Can fatigue be ruled out? If yes, explain how.**

Fatigue can be ruled out as the primary cause of failure in this case. Fatigue failure typically occurs in materials subjected to repeated cyclic loading over an extended period, which leads to crack initiation and propagation. The text does not mention any cyclic loading or stress history that would be characteristic of fatigue failure. Instead, the primary cause of failure is identified as pitting corrosion due to the corrosive environment, as described in earlier sections.

- **Can environmentally assisted failure be ruled out? If yes, explain how.**

Environmentally assisted failure, which includes mechanisms like stress-corrosion cracking (SCC) or hydrogen embrittlement, cannot be ruled out based on the information provided in the text. The text does mention the presence of chloride ions in the water, which can contribute to the corrosion process and potentially lead to SCC or other environmentally assisted failure mechanisms. However, while chloride ions are mentioned, the text does not explicitly state that SCC or hydrogen embrittlement were identified as contributing factors to the failure. Further analysis or testing would be needed to confirm or rule out these mechanisms definitively.

D. Recommendations to prevent similar failures in the future

- **How should the design, material, use, etc. be developed to avoid similar failures in the future? Provide several alternatives and indicate most promising.**

1. Material Selection: Consider using more corrosion-resistant stainless steel alloys, such as 1.4462, which demonstrated better corrosion resistance in the tests. This could enhance the tank's longevity in a corrosive environment.

2. Coatings and Linings: Apply corrosion-resistant coatings or linings to the inner surface of the tank. This protective layer can act as a barrier between the corrosive water and the tank material, significantly extending the tank's lifespan.

3. Water Treatment: Improve the water treatment process to reduce chloride ion content or other corrosive elements in the water. Maintaining water quality can minimize the aggressive nature of the environment.
4. Regular Inspection and Maintenance: Implement a routine inspection and maintenance program for the tank. This includes regular checks for signs of corrosion, cracks, or pitting. Address any issues promptly to prevent further deterioration.
5. Temperature Control: Maintain strict control over the temperature of the stored water. Lowering the water temperature, if possible, can reduce the corrosive effects on the tank material.
6. Stress Analysis: Perform stress analysis to ensure that the tank's design can withstand the combined effects of hydrostatic pressure, temperature fluctuations, and mechanical stresses, especially in areas where pipes or equipment interact with the tank.
7. Material Compatibility: Ensure that all materials used in the brewery's water system are compatible with the water chemistry. Consider materials that are less susceptible to corrosion in the presence of chloride ions.
8. Monitoring and Alarms: Install monitoring systems that can detect changes in tank conditions, such as temperature, pressure, and corrosion rates. Implement alarms to alert operators to potential issues before they lead to catastrophic failures.

Paper3_edit.pdf

1. Introduction

The subject of the analysis was the broken blade of cutter Ku 500VX that was used in meat processing. The aim of the study was to determine the failure cause and to give an answer how it could be avoided. General view of broken blade with marked area of the damage is shown in [Fig. 1](#). The performed tests included hardness measurements, fractography analysis and metallographic studies using stereoscopic, light and scanning electron microscopes.

2. Material and methods

Macroscopic analysis of outer surface and fracture of the blade was performed using stereoscopic microscope SMT 800 and scanning electron microscope JEOL JSM 5800 LV.

Examination on the cross-section of the blade was performed in non-etched state and after etching with reagent Mi19Fe containing 3 g of ferric chloride, 10 cm³ hydrochloric acid and 90 cm³ of ethanol according to polish standard PN-H-04503:1961P (what corresponds to reagent no. 26 according to international standard ASTM E407-07) using metallographic light microscope Epiphot coupled with Nikon digital camera. The paper presents a photographic documentation of characteristic structures.

Hardness measurements were made on the outer surface of the blade. The study was performed with Vickers method under the load of 10 kg (98.070 N) in accordance with standard DIN EN ISO 6507-1:1999 using Zwick hardness tester 321, running time was 15 s.

A general view of the broken blade is shown in [Fig. 1](#). Sample for testing was taken near to damage from the marked area ([Fig. 1](#)). The broken blade was made of high-speed tool steel typically used for cutting tools. The structure was made of



Fig. 1. General view of the broken blade. The arrow indicates the place where sample was taken for testing and indicates the place where the inscription (number "1") was made with engraving tool.

martensite matrix with precipitates of fine secondary carbides and undissolved primary carbides (with a light intensity of banding). No tendency to form carbides grid was observed ([Figs. 2 and 3](#)). This is a typical structure for properly heat treated high speed tool steels. The average hardness value was 456 HV10 (46 HRC).

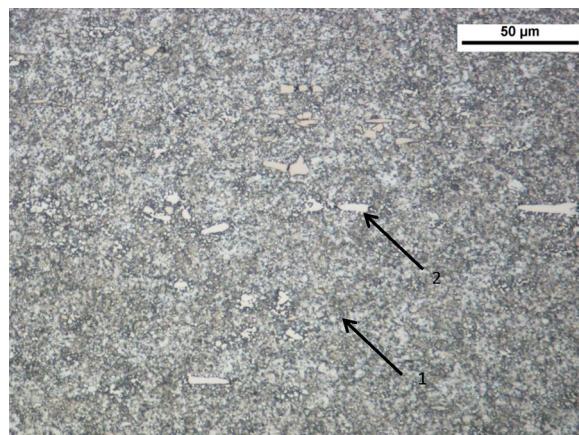


Fig. 2. Structure of martensite matrix with precipitates of fine secondary carbides [1] and undissolved primary carbides [2]. Light microscopy, etched with Mi19Fe.

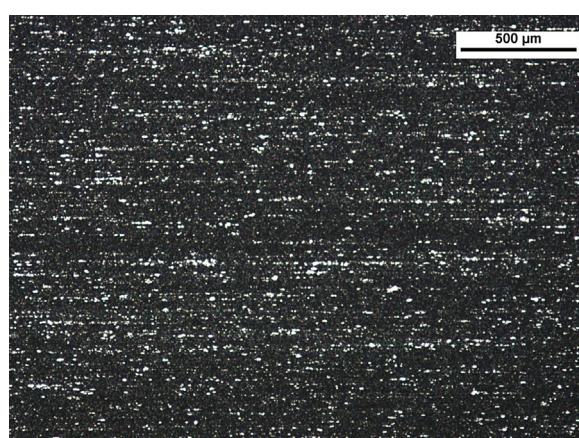


Fig. 3. The magnified part of area presented in [Fig. 2](#). Structure of martensite matrix with precipitates of fine secondary carbides. No tendency to form carbides grid was observed. Light microscopy, etched with Mi19Fe.

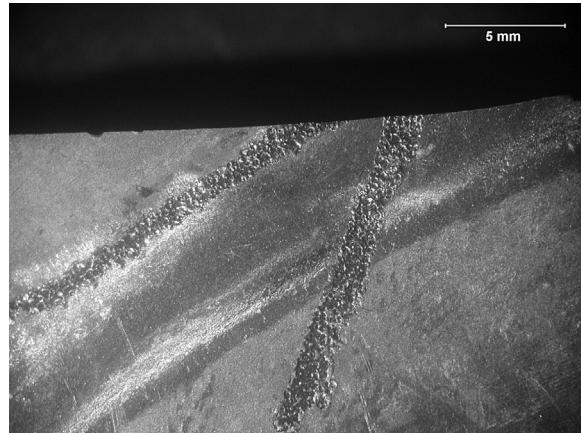


Fig. 4. The magnified part of area shown in Fig. 1. Place where the inscription (number "1") was made with engraving tool. Stereomicroscopy, non-etched state.

3. Failure analysis

3.1. Results of macroscopic examination

On the outer surface of the blade (Fig. 1), around the fracture, it was found an inscription – number “1”. This inscription was probably burnt during manual process with any sort of electric engraving tool used for marking materials or components (Fig. 4).

Additional macroscopic examination of the blade's fracture shows features of the fatigue fracture. Origin of the fracture is in place where the inscription was burnt (Figs. 5 and 6). Around the origin of the fracture, in place of burnt inscription, there were found cracks and changes of the microstructure (Figs. 7 and 8).

3.2. Results of microscopic examination

In non-etched state around the origin of the fracture it was found uneven surface with micro cracks (Figs. 9 and 10). On the outer surface of the specimen, around the origin of the fatigue fracture where the inscription number “1” was made, it was observed change in the structure due to local heating of the material till melting point (Figs. 11–13). This local heating of the material took place during engraving process. Additionally, the features of micro dendritic structure formed during quick cooling of liquid metal were visible (Fig. 13).

4. Discussion

The quality of the surface of cyclically loaded components is very important. Many observations confirm that the root cause of the micro cracks (causing the fatigue fracture) are primarily a surface's defects appearing during production process [1]. Studies of fatigue fracture surfaces provide a lot of findings concerning loading of the broken component. On that basis, the type and direction of the forces acting on the part can be shown and a general indication of the length of time from

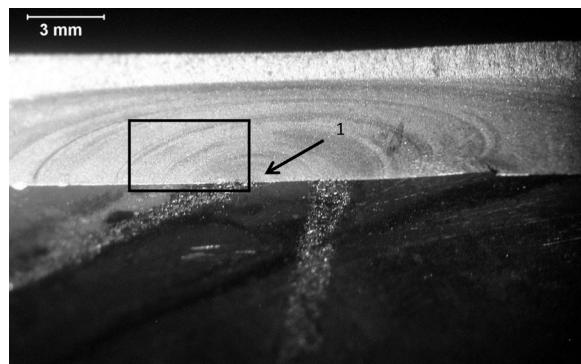


Fig. 5. Fatigue fracture with beach marks and origin [1] in place where the inscription (number "1") was made with engraving tool. Stereomicroscopy.

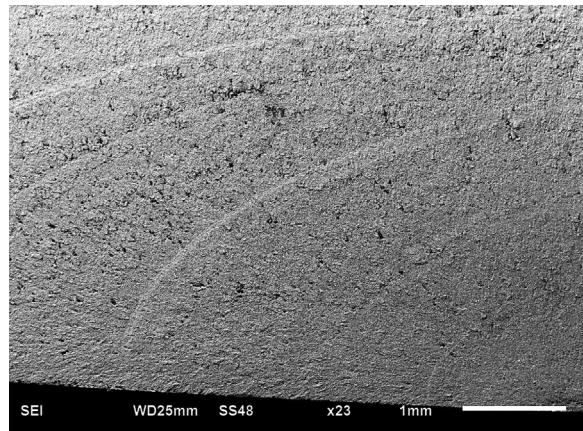


Fig. 6. The magnified part of area shown and marked in [Fig. 5](#). Fatigue fracture with origin and beach marks. Scanning electron microscopy.

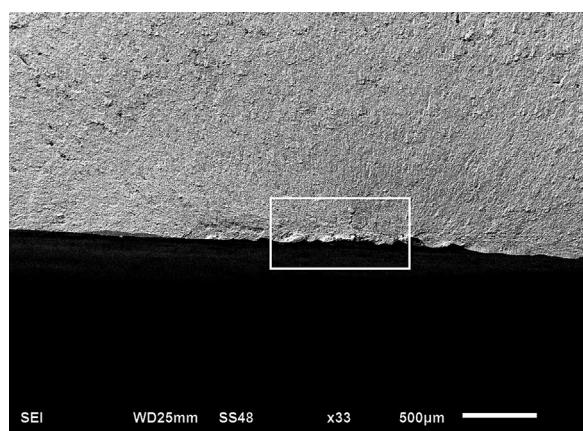


Fig. 7. Origin of the fatigue fracture. Scanning electron microscopy.

initiation to final fracture can be given [2]. In the same way the analysis of the fatigue fracture of examined broken blade, the location of the fracture's origin and beach marks, led to define the reasons of its damage.

The main cause of the damage of examined blade was engraving the inscription (number "1") with electric engraving tool. The burning of this inscription led to a local changes in the structure and to turn of micro cracks during service life of the

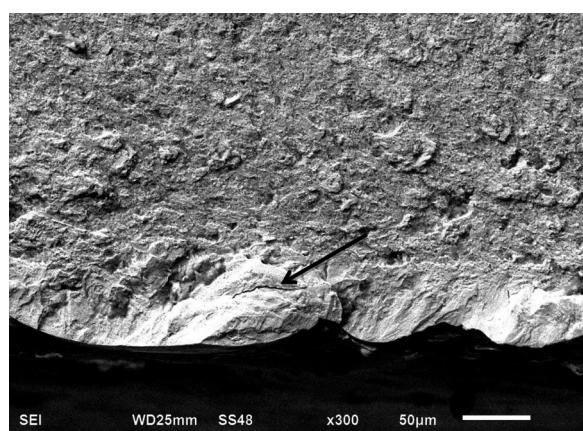


Fig. 8. The magnified part of area marked in [Fig. 7](#). Origin of the fatigue fracture. The arrow indicates the micro crack. Scanning electron microscopy.

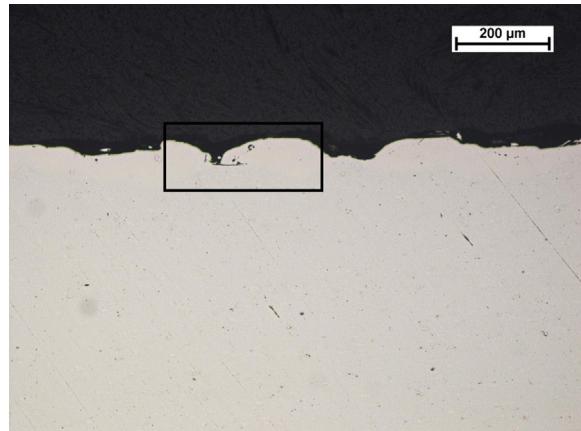


Fig. 9. Surface of the specimen around the origin of the fatigue fracture with visible micro cracks. Cross-section of the blade. Light microscopy, non-etched state.

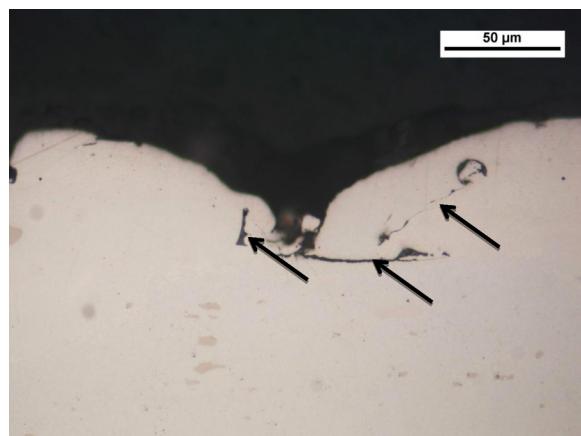


Fig. 10. The magnified part of area marked in **Fig. 9**. Surface of the specimen around the origin of the fatigue fracture with visible micro cracks indicated with arrows. Light microscopy, non-etched state.

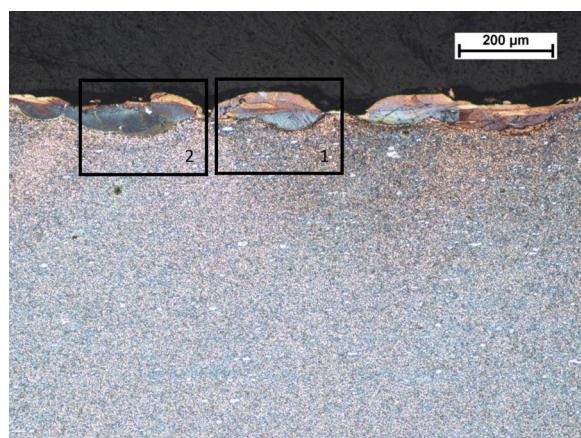


Fig. 11. Changes in the structure of the specimen's surface in the place where the inscription (number "1") was made with engraving tool. Cross-section of the blade. Light microscopy, etched with Mi19Fe.



Fig. 12. The magnified part of area marked with no. 1 in Fig. 11. Changes in the structure caused by local melting of the specimen's surface where the inscription was made with engraving tool. Light microscopy, etched with Mi19Fe.



Fig. 13. The magnified part of area marked with no. 2 in Fig. 11. Changes in the structure caused by local melting of the specimen's surface where the inscription was made with engraving tool. The arrow indicates the micro dendrites formed during quick cooling process of melted material. Light microscopy, etched with Mi19Fe.

blade what provided to fatigue fracture. In case of examined blade electrical engraving process caused local heating of the material in the surface. The temperature level was high and caused local melting of the material in the surface. This led to changes of the structure and form the micro dendrites. As a result of these changes the stresses (already established residual stresses) occurred in surface what provided to micro cracking. The same defects are very often observed during EDM (electrical discharge machining) that is often used during the production of components made by tool steels [3].

Marking of material and components are common used by manufacturers. During this process, especially such sort of engraving when much heat is emitted (for example electric engraving), the surface is affected what can provide to effect of structural notch. Even laser engraving or stamping can be detrimental to a component's fatigue life [4,5]. Marking should not be made on highly stressed areas, near edges or on sensitive seal surfaces. High-speed steel tools are mainly produced by single companies and in smaller sizes. Due to this an introduction of automated engraving process (in order to obtain better control parameters) may not be cost effective. For components made by tool steel to provide its traceability and to avoid reducing of fatigue strength it is recommended to use more safety identification marks such as adhesive labels.

588962

Materials safety: article exercise 1

The subject of investigation was a broken blade used in meat processing made from heat treated steel typically used in tool making. To analyze the blade, the outer surface, cross-section and hardness were tested. Macroscopic analysis was performed using both stereoscopic and scanning electron microscopy. The cross-section was analyzed using a metallographic light microscope in both non-etched and etched states. Hardness of the material was measured with the Vickers method. All test methods were in accordance with relevant standards of the field. The results were presented in graphical form as figures.

During the analysis, a carved figure of "1" was found on the blade in the fractured area. This was most likely done using an electrical engraving tool that heats up the material rapidly. After engraving, the blade was likely cooled down rapidly by dipping it into water. The process caused changes in the microstructure of the blade and lead to micro cracks appearing on the blade, causing a fatigue fracture. The fatigue fracture was confirmed during macroscopic analysis with the starting point identified at the carved figure. With such overwhelming and clear evidence, alternative failure mechanisms can be ruled out, although the report fails to address them.

To prevent such failures from happening, the durability of the part should be ensured under design conditions. In this case, the carved figure was done after processing most likely to number the part. If such numbering is required, it should be done using a method that doesn't affect the structure of the material.

477882

Materials safety: article exercise 1

Material and methods

The performed tests included hardness measurements, fractography analysis and metallographic studies using stereoscopic, light and scanning electron microscopes. The macroscopic analysis of outer surface and fracture of the blade of cutter Ku 500VX (high-speed tool steel) was performed with stereoscopic microscope SMT 800 and scanning electron microscope JEOL JSM 5800 LV. The cross-section of the blade was examined in non-etched state and etched state with reagent Mi19Fe (3 g of ferric chloride, 10 cm³ hydrochloric acid and 90 cm³ of ethanol) (polish standard PN-H-04503:1961P, corresponds to reagent no. 26 in international standard ASTM E407-07). Footage from the light microscope was captured with Nikon digital camera. Hardness was measured on the outer surface of the blade based on Vickers method under the load of 10 kg (98.070 N) (DIN EN ISO 6507-1:1999) using Zwick hardness tester 321 with a running time of 15 s. The average measured hardness value was 456 HV10 (46 HRC). The sample near the damaged area showed that the material structure was martensite matrix with precipitates of fine secondary carbides and undissolved primary carbides with a light intensity of banding. No carbides grid was found which is typical heat-treated high-speed tool steels.

Failure analysis

In macroscopic examination inscription of a number 1 was found at the fracture. The number was probably suggested to be made electric engraving tool for marking materials. Examination also found fatigue fractures with cracks and changes in microstructure at the origin of fracture around the inscription.

In microscopic examination microcracks were found on the uneven surface at origin of the fracture. Material structure had also changed around the inscription due to the extensive local heating up to melting point of the material during the engraving process. When the melted metal had cooled and solidified, micro dendritic structure was formed.

The study found that the primary cause of the failure was engraving the inscription of the number 1 with an electric engraving tool. The heat, melting and solidifying of material in engraving process caused local changes in material structure and micro dendritic structures. Cutting blades require high surface quality due to the cyclic loads under use. It has been confirmed that common cause of micro cracks leading to fatigue fractures have been surface defects of the products during production which led to microcracks. In this case, the local changes of structures on the surface caused additional stresses leading to microcracks and eventually fatigue failure of the blade when it was used.

Ruling out alternate failure mechanisms

Typical characteristics of cutting tool materials are hardness (tool must be harder than the cut material, abrasive wear resistance), toughness (tool must stand discontinuous cutting and variation of chip thickness), heat resistance (tool's mechanical properties must stand cutting temperature), low friction (low friction between tool and workpiece reduces wear and improves surface quality) and low chemical reactivity (tool must not react chemically with workpiece or air). High speed steel is commonly used due to good hardness, high toughness and good heat resistance.

Plastic deformation can happen around the cutting edge of cutting tools due to the high cutting temperatures and mechanical loads which cause softening of the material. The study didn't mention any major discoloration of tool (due high temperatures) or plastic deformation around failure so plastic deformation causing a crack could be ruled out.

Creep is time-dependent deformation caused by high temperatures and constant stress. It can cause change of tool geometry in high temperatures leading to poor tool performance, earlier tool changes, but the cracks of this case shouldn't be related to creep.

Brittle fracture happens suddenly under heavy loads. High speed steel should resist these kinds of failures, but bad quality tool or improper usage of tool (excessive load) could cause brittle fracture.

Fatigue is a typical failure mechanism for parts under cyclic load. Cyclic loads happen in machining causing fatigue cracks, so fatigue failures are possible.

Environmentally assisted failure can happen due to oxidation and corrosion in high temperatures. The study didn't mention any signs of environmental effects on tool, so this mechanism could be ruled out.

Prevention of similar failures in the future

Marking of material and products is a common and often required process for identification in manufacturing. Typical methods, such as EDM and laser will cause changes in surface structures that can lead to early fatigue failures. Cutting tools are also typically hard materials, which will also reduce options for available methods for engraving.

It is recommended to use other marking methods such as adhesive labels that are placed away from areas of the tool that will have any wear in use. Labels and adhesives should also withstand the usage of cutting fluids so that the labels will remain attached.

If the engravings must be made, then they should not be placed on highly stressed areas, near edges or on sensitive seal surfaces. This will reduce the possibility of early fatigue failure and increase the tool life.

Additional research possibilities

From manufacturing point of view, it would have been important to know more about the usage of the tools, such as machine, machining method, machining parameters, workpiece material and how was the chip formation and surface quality before failure to rule out the possibility of unsuitable cutting parameters and machine or operator failure during the machining.

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Materials safety: article exercise

The report analyses the cause of a broken blade made from high-speed tool steel for meat processing. The investigation was carried out with hardness test, fractography, and metallographic studies using stereoscopic, light and scanning electron microscopes (SEM). The macroscopic analysis examines the outer surface with stereoscopic and SEM. The microscopic examines the cross-section in both etched and non-etched state using a metallography light microscope. The hardness measurement was done on the outer surface with Vickers method and the hardness obtained has a value of 456 HV10. The results for macroscopic analysis show that, on the outer surface, there is an inscription (number "1") and features of fatigue fracture can also be found. On the microscopic scale, the analysis shows that the overall material composition appears to be normal, however, structure changes and microcracks can be found around the origin of the fracture.

The primary cause of the failure comes from the electric engraving of the inscription. During the engraving process, the material on the outer surface was heated locally, leading to local melting and changes of the structure. When the tool cooled down, surface defects were formed, resulting in residual stresses in the affected areas. Due to cyclic loading during operation of the tool, the stresses near these affected areas became higher than the designed values, and microcracks started to form and propagate. Finally, the blade fractured.

It can be concluded from the report that the main reason for the failure of the blade involves fatigue and brittle fracture. The traces of fatigue microcracks observed by the analysis are the main causes of fatigue failure. In addition, the blade made from high-speed tool steel, whose properties include high hardness and brittleness, means that the blade might have experienced sudden brittle fracture. Other mechanisms such as plastic deformation and creep could be ruled out in this case. If the tool was designed and used properly, there should be no plastic deformation involved. In addition, according to the microscopic analysis, the grain structures of the brittle material were normal. Thus, the blade is not likely to deform plastically. In general, fatigue failure can be assisted by the environment of operation. However, it is required more data and information to include the environmental effects, which is not provided in the report. If it can be assumed that the working environment of the blade had negligible contributions to the fatigue, then environmental factors can be ruled out from the main cause of the failure.

To prevent similar accidents, the report suggests avoiding engraving or marking highly stressed areas, or sensitive surfaces. Other practices can include considering surface damages and accounting for fatigue strength during the design phase. However, this approach is not feasible and economically efficient for this type of product. It is more feasible to educate the material manufacturers about the effects of engraving to failure of material and how to use engraving safely. It is still risky to continue with engraving processes as any engraving method that introduces high



heat to the material might cause similar unexpected problems. The most prominent approach is to use alternative labeling method without damaging the surface.

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Materials safety: article exercise

You are working as a materials expert in your organization and your responsibility is to guarantee safe and efficient use of materials in your facility. One day, a failure in similar facility is brought to your attention, and you need to investigate the possible implications this failure has for your facility. Your job is to interpret and analyze the given failure report and to write a report, which will allow others in your organization to understand the key developments and causes leading to the failure and the necessary actions for prevention of such failures.

Read the given article and analyze the failure. Describe, how the deformation and failure mechanisms presented during the course are reflected in the case and establish the chain of actions leading to the failure. The report should work as an introductory material to your team; it should not be very long but it should enable other team members to understand the key features of the failure without reading the failure report itself.

In addition to establishing the primary cause of the failure, show why alternate failure mechanisms can be ruled out. Some failure mechanisms have not been discussed in the course yet. Conduct the analysis using your present knowledge on the subject.

If the author of the failure analysis has, in your view, neglected to address some aspects of the failure, you may indicate this in your report and suggest tests or actions that should have been done to clarify the issue.

Prepare your response by editing this word document and export it as PDF. The file name identifies you and the article. Do not change the file name (other than the extension to pdf). E-mail the pdf to "materials.safety@iikka.fi".

You may use the question list below to guide you in your analysis:

- A. Description of investigation methods applied
 - What means of investigation were used in the failure analysis?
 - What computational methods were used?
 - What material or results were obtained?
- B. The primary cause of the failure and description of the failure mechanism
 - What is the primary cause of the failure (also provide reasoning)?
 - What's the chain of action that led to the failure?
- C. Ruling out alternate failure mechanisms
 - Can plastic deformation be ruled out? If yes, explain how.
 - Can creep be ruled out? If yes, explain how.
 - Can brittle fracture be ruled out? If yes, explain how.
 - Can fatigue be ruled out? If yes, explain how.
 - Can environmentally assisted failure be ruled out? If yes, explain how.
- D. Recommendations to prevent similar failures in the future
 - How should the design, material, use, etc. be developed to avoid similar failures in the future? Provide several alternatives and indicate most promising.

Analysis

The case is about a blade from a meat cutter, that broke at the edge. To investigate the failure a macroscopic analysis of the surface was performed with a stereoscopic and a scanning microscope. In addition, an examination of the cross-sectional area was done in an etched and non-etched stage with a metallographic light microscope. A hardness measurement was also done but did not show a value out of the ordinary.

In the macroscopic analysis it was observed, that the failure appeared around the inscription "1" of the blade. The "1" was probably electrical engraved into the blade. There were also signs of fatigue failure and the origin seems to be at the inscription, at the origin were cracks. The microscopic analysis on the non-etched part showed microcracks and an uneven surface near the inscription.

In the etched analysis the pictures show a different microstructure around the inscription due to the local heating, when making the inscription.

All observations point to the conclusion, that the engraving of the inscription is the main cause for the failure. During the engraving process, heat was induced into the blade at specific points leading to a change in the microstructure and surface. During the service life, microcracks were forming at the changed parts. These cracks lead to the fatigue failure after some time.

The failure seems clear, since all analysis support the theory and there is no hint, that it could have been a different failure mechanism.

To avoid this kind of failure, it is important to try not to weaken the part through for example engraving. A different marking method, which not induces heat should be preferred. Moreover, the marking should be done at a less critical point.