CASE HISTORY—PEER-REVIEWED

Marine Atmospheric SCC of Unsensitized Stainless Steel Rock Climbing Protection

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Abstract A failure investigation into the root cause of fixed austenitic stainless steel climbing anchor hardware in tropical marine climates has been presented. The incident 316L climbing anchor was fixed in a seaside limestone cliff in southern Thailand and underwent transgranular chloride stress corrosion cracking (TGSCC) after 10 years of service. Since stainless steel does not normally undergo stress corrosion cracking (SCC) at ambient temperatures, the conditions known to promote ambient temperature TGSCC of austenitic stainless steel are reviewed. A mechanism that may give rise to TGSCC in limestone climbing anchors in warm marine environments is postulated.

Keywords Austenitic stainless steel · Corrosion failure analysis · Stress corrosion cracking · Failure mechanism

Introduction

In the last decade, rock climbing on limestone cliffs in warm marine environments such as southern Thailand has grown in popularity. As a result, numerous stainless steel bolts and hangers were installed to serve as fixed climbing protection (Fig. 1). Typical fixed hardware for rock climbing consists of a stainless steel expansion bolt, nut, and a 316L hanger (Fig. 1), where the hanger allows for convenient attachment of a quickdraw (i.e., two aluminum

(Al) carabiners connected by a nylon sling) through which a nylon climbing rope can be clipped. Soon after installation into seaside, limestone cliffs, such as in southern Thailand, the stainless steel hardware became stained with rust and stress corrosion cracking (SCC) was reported [1].

A fractured hanger fragment segment (Fig. 2) from a limestone cliff at Railay Beach (Fig. 3) in the Krabi Province of southern Thailand was available for analysis. Geologically, the limestone cliff is tower karst, part of the Ratburi group [2]. The hanger was reportedly installed in 1994, approximately 15 m above a cliff base that contacts the ocean at high tide. In 2004, the 316L hanger fractured when tested by hand and a fragment recovered for analysis. Since SCC of unsensitized austenitic stainless steels does not normally occur at ambient temperature in chloride (Cl) environments at near neutral pH such as seawater or during marine atmospheric exposures [3–29], a preliminary study was undertaken to determine the type of SCC that occurred. The purpose of this study was threefold: determine the type of SCC occurring in the 316L material; compare this SCC failure with other rare reports of ambient temperature transgranular chloride stress corrosion cracking (TGSCC) incidents, particularly with laboratory studies conducted as follow-up work to SCC failures in indoor swimming pool atmospheres [19-21, 30, 31]; and to evaluate two titanium (Ti) materials, unalloyed and Ti-6Al-4V, currently used in place of stainless steel at coastal climbing areas.

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Experimental

Chemical analysis of stainless steel and titanium alloys was conducted using standard emission/plasma spectroscopic and combustion techniques. Metallography was conducted using standard metallographic procedures for stainless



Fig. 1 Photograph of typical rock climbing bolt used for fixed protection an austenitic stainless steel expansion bolt, nut, and 316L hanger (most common). Dashed lines indicate fragment recovered after failure and available for analysis

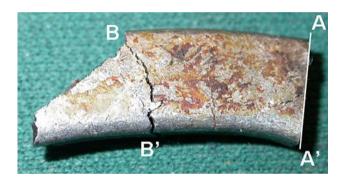


Fig. 2 Fragment of hanger after 10 years at Railay Beach in Krabi Province, Thailand. Section A–A' shows area sectioned for metallography. Fracture B–B' shows pre-existing fracture at which the sample was broken open for SEM/EDS examination

steel. Microhardness tests were performed on metallographic mounts using a Vickers scale. X-ray photoelectron spectroscopy (XPS), also known as electron spectroscopy for chemical analysis (ESCA), was performed on corrosion product with a PHI Quantum 2000 instrument using Al as an X-ray source.

Elemental composition of solid (corrosion) product was also obtained using energy dispersive spectroscopy (EDS). Boiling magnesium chloride (MgCl₂) SCC resistance tests were run according to ASTM G36 [32]. An exemplar hanger from the same manufacturer was used for chemistry and hardness testing (Petzl, 10 mm). The hanger was rated to 25 kN (5620 lbf).

Results

A cut along Line A-A' shown in Fig. 4 was made to expose a transverse cross section of the hanger fragment. Extensive cracking through the hanger thickness was observed

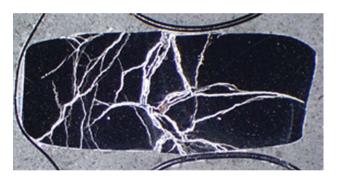
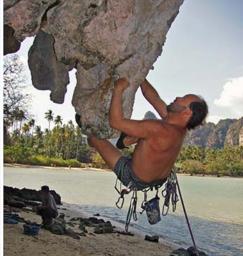


Fig. 4 Stereomicroscope view of hanger transverse cross-section (Section $A-A^\prime$, shown in Fig. 2)

Fig. 3 Photographs of limestone formations (tower karst) near Phranang and Railay Beach in the Krabi Province, Thailand, a popular rock climbing area. Photographs reprinted with permission from Karl Bralich at peaklightimages.com







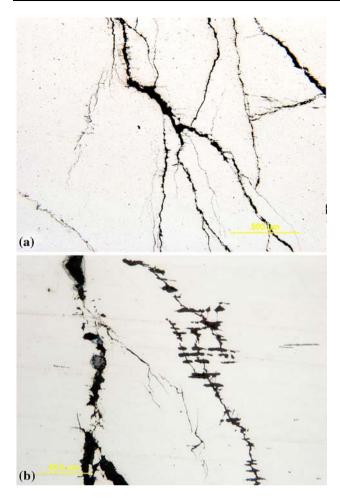


Fig. 5 Optical micrographs of unetched microstructure (Section A–A', shown in Fig. 2)

when examined with a stereomicroscope. Optical micrographs of the unetched microstructure (Fig. 5a) show the presence of highly branched cracks. Higher magnification showed that the cracks tend to follow manganese sulfide (MnS) inclusions (Fig. 5b). An optical micrograph of the etched microstructure clearly shows that the failure was a result of TGSCC (Fig. 6).

The fragment was broken open along the large, pre-existing crack (labeled B–B') as shown in Fig. 2. A photograph (Fig. 7) and a SEM micrograph (Fig. 8) of the fresh fracture surface are provided. EDS of the corrosion product on the freshly exposed surface (Fig. 8) shows extremely high levels of magnesium (Mg) compared to the 316L alloy elements of iron (Fe) and chromium (Cr). Smaller amounts of chlorine (Cl), sulfur (S), potassium (K), and calcium (Ca) were also observed. XPS/ESCA results (Table 1) of the deposits on the freshly exposed fracture surface, after sputtering to a depth of between 50 and 100 Å, also showed high levels of Mg as well as Cl, sodium (Na), and Ca. A substantial amount of nitrogen (N) was found as well. This may indicate the presence of



Fig. 6 Optical micrograph of etched microstructure (Section A-A', shown in Fig. 2)

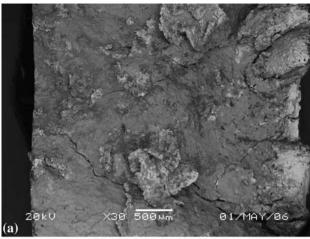


Fig. 7 Photograph of fresh fracture surface generated by breaking open along pre-existing cracks in hanger (Fracture B–B', shown in Fig. 2)

ammonium, nitrites, or nitrates. The Mg level of the near-surface corrosion product, as detected by XPS/ESCA, was ten times higher than the Ca levels, six times higher than the Na levels, and five times higher than the Cl levels. These cation/anion ratios are distinctly different from seawater, where Na levels are typically eight times higher than Mg, Mg five times higher than Ca, and Cl 15 times higher than Mg [33, 34].

The fracture surface was then cleaned by sonication in isopropanol. An SEM image of the cleaned fracture surface shows transgranular fracture (Fig. 9). Cl was still detected on the fracture surface after cleaning. Since there was insufficient material available for chemical or hardness tests, an exemplar stainless steel hanger of the same brand





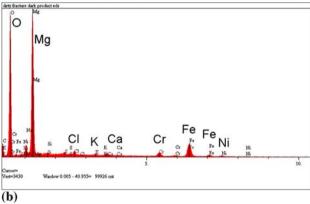
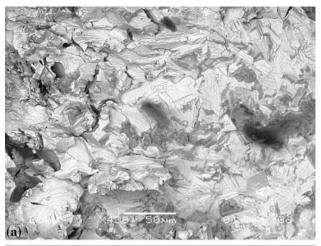


Fig. 8 (a) SEM micrograph of fresh fracture surface prior to cleaning, Fracture B–B'. (b) EDS spectrum of fresh fracture surface prior to cleaning, Fracture B–B', showing high levels of magnesium (Mg): O, oxygen; Cl, chloride; K, potassium; Ca, calcium; Cr, chromium; Fe, iron; and Ni, nickel

Table 1 Atomic concentrations (in %)^a of elements at surface using XPS-ESCA

Element	Concentration (%)
Carbon (C)	52.4
Oxygen (O)	35.4
Magnesium (Mg)	3.0
Iron (Fe)	2.6
Nitrogen (N)	2.2
Silicon (Si)	0.8
Chromium (Cr)	0.8
Nickel (Ni)	0.7
Chlorine (Cl)	0.6
Sodium (Na)	0.5
Aluminum (Al)	0.4
Calcium (Ca)	0.3
Sulfur (S)	0.2
Phosphorus (P)	0.1

 $^{^{\}rm a}$ Normalized to 100% of the elements detected. XPS does not detect hydrogen (H) or helium (He)



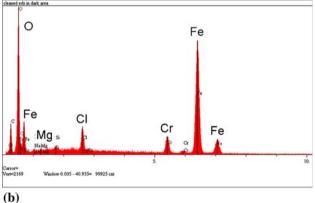


Fig. 9 (a) SEM micrograph of SCC fracture surface after cleaning showing transgranular fracture, Fracture B–B'. (b) EDS spectrum of SCC fracture surface after cleaning showing transgranular fracture, Fracture B–B', showing high levels of chloride (Cl): O, oxygen; Mg, magnesium; Cr, chromium; and Fe, iron

Table 2 Hardness values for climbing anchors

Material	Hv
316L Hanger, incident	A
316L Hanger exemplar	350
Welded cold-shut, unalloyed titanium	180
U-Bolt, Ti-6Al-4V	336

Note: A, insufficient material to determine hardness

(Petzl, 25 kN, 10 mm) was purchased and examined. The hardness of this exemplar hanger was 350 Hv, consistent with a high degree of cold work in the rolled material (Table 2). Chemical analysis was consistent with 316L stainless steel (Table 3). The chemistry and hardness of the other two materials, a welded titanium glue-in bolt (10 mm diameter) and a titanium glue-in U-bolt (7.9 mm diameter), were determined (Fig. 10). The alloy chemistry of the welded titanium glue-in bolt was consistent with unalloyed titanium and the U-bolt alloy chemistry was consistent with



Table 3 Chemical analysis of exemplar 316L hanger (wt.%)^a

	Carbon (C)	Chromium (Cr)	Nickel (Ni)	Manganese (Mg)	Silicon (Si)	Phosphorus (P)	Sulfur (S)	Molybdenum (Mo)
Hanger	0.018	16.47	10.17	1.80	0.61	0.024	< 0.005	2.11

^a Balance iron (Fe)

Table 4 Chemical analysis of titanium anchors (wt.%)^a

	Aluminum (Al)	Vanadium (V)	Iron (Fe)	Oxygen (O)	Carbon (C)	Nitrogen (N)
Welded cold shut	0.40	< 0.005	0.053	0.078	0.005	0.012
U-Bolt	6.4	3.73	0.053	0.078	0.005	0.012

^a Balance titanium (Ti)



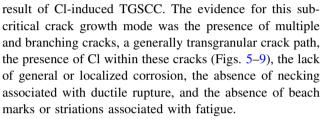
Fig. 10 Photographs of glue-in titanium bolts used for fixed climbing protection; unalloyed welded titanium (top) and Ti-6Al-4V U-bolt (bottom)

Ti-6Al-4V chemistry (Table 4). Hardness of the titanium bolts was 180 and 336 Hv, respectively (Table 2).

Boiling MgCl₂ tests [32] were run on the exemplar 316L hanger, the welded glue-in titanium and Ti–6Al–4V U-bolts. No external loads were applied to any of the materials. The unloaded stainless steel hanger was found to crack in under 4.5 h in 43% MgCl₂ (155 °C) and under 26 h in 35% MgCl₂ (125 °C). In both situations, the cracks occurred at the 90° bend. As expected, neither the unalloyed welded titanium glue-in nor the Ti–6Al–4V glue-in U-bolt showed any evidence of SCC after 670 h in boiling 35% MgCl₂ [35].

Discussion

The SEM and optical metallography clearly showed the failed stainless steel hanger from Thailand cracked as a



The transgranular crack morphology suggested that the field fractured stainless steel hanger had not undergone any sensitization, i.e., annealing in a temperature range or for a time that could result in Cr depletion at grain boundaries which would lead to intergranular crack morphology. The observed Cl-induced TGSCC at ambient temperatures is in conflict with the generally held belief that temperatures above 60 °C are required for SCC of unsensitized austenitic stainless steels [4, 5, 9, 10, 15–18, 22–24, 26–29]. Previous studies showed that the 300-series alloys do not generally exhibit SCC in marine atmospheric environments in spite of being stressed to 90% of their tensile yield strengths and a 452-day exposure [10, 15, 36, 37]. In addition, marine atmospheric exposure tests performed at Kure Beach, NC, on various austenitic stainless steel coupons showed no signs of SCC after 26 years [3].

As a result of its good performance at ambient temperature, austenitic stainless steels are widely used in seawater, food services, architectural, and swimming pool applications without incident. Conditions thought to produce SCC in unsensitized austenitic stainless steels are a combination of: temperatures elevated above 60 °C; nearly saturated halide solutions; or tensile stress, either residual or applied [23]. Of these factors, temperature is generally considered to be the most important.

Stress Effect

The mechanical loads responsible for the TGSCC observed were clearly a result of the residual stresses imposed at the time of manufacture and not a result of service-imposed loads, which are a very small fraction of time on the rock



cliffs. This conclusion is substantiated by the fact that the unloaded, exemplar hanger cracked rapidly in only 4.5 h when exposed to the boiling MgCl₂, a short time when compared to times-to-failure for 321 specimens in boiling 42% MgCl₂ [22]. Technical literature from as far back as the late 1800s clearly shows that residual stresses from metal forming operations are sufficient to cause SCC [7, 38–41]. The hanger was heavily cold-worked as shown by the 350 Hv of the exemplar hanger (approximately 50% cold work) [42]. Typical annealed 316L material has a 230-Hv hardness [43].

Comparison with Literature Examples of Ambient Temperature SCC of Austenitic Stainless Steels

Although it is a generally held belief that SCC of stainless steel only occurs above 60 °C in relatively benign environments such as marine atmospheres, a survey of the literature turned up a few key examples where SCC was observed below this temperature:

- Sensitized stainless steel has been reported to undergo intergranular SCC (IGSCC) at temperatures as low as 20 °C [12, 17, 24, 44].
- 2. Iron contaminated Type 304 stainless steel components exposed to marine atmospheric conditions have been reported to undergo TGSCC [8, 45, 46].
- 3. High sulfur, free machining Type 303 stainless steel stressed to 90% of the yield strength was found to exhibit TGSCC in the Mont Blanc tunnel (linking France and Italy) where samples were exposed to road salt, soot, tire rubber, and automotive exhaust. Type 304 stainless steel samples did not crack [47].
- 4. Creviced conditions can generate TGSCC on stainless steels exposed to coastal atmospheres [11]. TGSCC has been noted also in the crevices formed by Type 304 nuts on threaded fasteners completely immersed in 3 wt.% NaCl (neutral pH) at 35 °C [9].
- 5. Ambient temperature SCC occurred on the outside surface of warm, Type 302 stainless steel pipes that may have been contaminated with residual flux [29]. Laboratory tests on flux-contaminated tubes thermally cycled in high and normal humidity atmospheres were able to duplicate the SCC. Isothermal tests confirmed that deposits of zinc chloride (ZnCl₂) or flux on stressed, Type 302 stainless steel pipe surfaces caused SCC after 5200 h (217 days) at 40 °C. The pH ranged from 2 to 6 [29]. No cracking was observed on NaCl-contaminated pipes.
- Unsensitized Type 304 stainless steel specimens, which were completely immersed in saturated MgCl₂ solutions, then withdrawn and allowed to completely

- dry under controlled relative humidity (RH), generated TGSCC at 30 °C [48]. Similar tests using saturated NaCl solutions did not crack.
- 7. There have also been a number of reports of stainless steel ceiling rods above indoor swimming pools that have failed from TGSCC [19–21, 30, 31, 49].

None of the first four incidents of ambient temperature SCC described above can be used to explain the Thailand hanger cracking since

- 1. the hanger was not sensitized and had not cracked intergranularly;
- 2. no iron contamination was found on the hanger;
- the hanger material was Type 316L stainless steel, and was not high sulfur stainless steel, such as Type 303; and
- crevice corrosion cannot be responsible for the TGSCC since cracking was observed on the hanger section that extended away from the rock surface (see Fig. 1).

A close examination of the conditions that lead to SCC in items 5–7 above, however, does show some similarities to the conditions of the subject hanger. These conditions and their possible similarities to the Thailand hanger require some discussion.

The most notorious ambient temperature TGSCC report listed in items 1–7 above occurred in indoor swimming pool ceiling rods. The unexpected failure in 1985 of Type 304 stainless steel rods used to suspend a concrete roof in an indoor swimming pool atmosphere in Ulster, Switzerland, resulted in the tragic death of 13 people. This accident was followed by numerous, well-funded laboratory tests to elucidate the SCC mechanism [19–21]. These types of failures occur only in the atmosphere above the swimming pool and do not occur if the steel is immersed in or is continually wet by the pool water. One of the mechanisms Oldfield postulated for the TGSCC was the evaporation and transport of chloramines, a volatile and unstable byproduct of chlorine with body fluids, is the most likely mechanism for transport of Cl from the swimming pool to the ceiling surfaces where they can decompose to acid chlorides (HCl) and accumulate to high levels. Analysis of corrosion products on failed wires showed complex and varied mixtures of Zn, Ca, and Al, possibly from construction materials, as well as Cl. Such a mechanism of chloramine transport to the stainless surface is unique to indoor swimming pool atmospheres and is unlikely to have affected the hangers.

The other mechanism postulated by Oldfield and Todd [21] to explain the ambient swimming pool failures was motivated by Shoji's work [30, 31] which indicated that SCC of Type 304 and 316 stainless steel U-bend specimens



Table 5 Equilibrium concentration of saturated Cl solutions at 25 °C [31]

Chloride	Saturated concentration, wt.% as Cl	Equilibrium RH of saturated Cl solutions (%)
Sodium chloride (NaCl)	16	75
Magnesium chloride (MgCl ₂)	27	33
Calcium chloride (CaCl ₂)	29	31
Zinc chloride (ZnCl ₂)	42	10

occurred from surfaces contaminated with various Cl salts including MgCl₂ exposed to certain fixed RH levels. Cracking was observed to occur at ambient temperature for MgCl₂, CaCl₂, and ZnCl₂ salts at fixed, low RH corresponding to the equilibrium humidity of a saturated solution of the salt in question (Table 5).

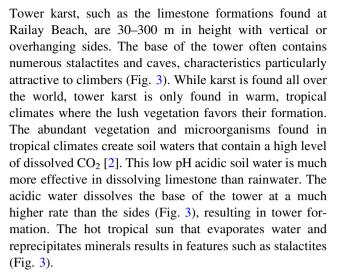
In Shoji's research, RH above the equilibrium humidity did not result in TGSCC since the additional moisture dilutes the saturated Cl film and makes them less aggressive. TGSCC was observed in specimen surfaces contaminated with MgCl₂ salt that were held in a closed vessel above a saturated MgCl₂ solution for 10,000 h (417 days) at 30 °C. For MgCl₂, the RH range for SCC at ambient temperature was 25–45%. A survey of indoor pool RH indicated that typical levels are in the range of 50–60% with some pool atmospheres having RH lower than this range.

In examining concentrated Cl solutions (Table 5), a saturated MgCl₂ solution delivers more chloride (27% Cl ion) as compared to saturated NaCl solutions (16% Cl ion). By way of comparison, seawater contains 1.9% Cl ion [34]. In the Shoji studies, no cracking was observed for NaCl, possibly because of the lower Cl content in a saturated solution [30, 31]. The Shoji studies are supported by Umemura et al. [48], which showed that MgCl₂, but not NaCl films, undergoing wet/dry cycles promotes TGSCC at temperatures as low as 30 °C in unsensitized Type 304 stainless steel.

Extremely high (saturated) Cl concentrations are needed to promote ambient temperature TGSCC. This, in turn, requires a relatively low RH and specific Cl salts such as MgCl₂ and ZnCl₂. Oldfield concluded [21] that the necessary high-salt/low-humidity combination rarely occurs outside of indoor swimming pool environments. For instance, the RH in marine environments, where Cl salts are generally found, tends to be too high, and Cl salts are not generally found in inland areas where the RH is lower.

Geology and Climate Considerations of Southern Thailand

The environment to which the hangers were exposed requires some discussion of the local geology and climate.



The cliff's proximity to the ocean (high tides reach its base) and its steep, overhanging nature allow high concentrations of Cl salt deposits to accumulate on the rock climbing hardware surface. When Ca- and Mg-carbonates leaches from the low pH waters in contact with limestone and mixes with the accumulated ocean salts, concentrated MgCl₂ and CaCl₂ solutions can form on the hanger surface. MgCl₂ and CaCl₂ deposits are deliquescent, attracting water from the atmosphere, which together with high humidity and the hot tropical sun can provide for frequent wet-dry cycles. The average RH levels recorded at the nearby seaside airport of Phuket, Thailand, are high. For example, the average RH level was 84.4% with an average temperature of 86.0 °F (30.0 °C) in 2001 [50]. However, the annual average RH and temperature do not indicate the range of conditions encountered. The average high and low RH levels in 2001 were 97.3 and 68.6%, respectively. The average low RH level varies with the season. For December 2000 and January and February 2001, the average low RH levels were 57.3, 57.1, and 55.0%, respectively, whereas in August, September, and October 2001, the average low RH levels were 76.7, 76.8, and 78.5%, respectively. Daily low RH levels below 50% are common in the winter months. The average high RH levels were between 95 and 99% for all 12 months in 2001. Notably many monthly average low RH levels were within the range of RH observed in indoor swimming pool atmospheres (50-60%) as reported by Oldfield [21]. Low RH appears to be more important than high RH for causing ambient temperature TGSCC for the salts present on the seaside tower karst, particularly MgCl₂.

Although the range of air temperatures and RH are known for the region, the incident hanger surface temperature was not known since metal surface temperatures were not available. Some metal surfaces exposed to the sun, such as roofing material, can get quite hot. For example, the maximum surface temperature of an unpainted metal roof



in Nashville, TN, was reported as 140 °F (60 °C) [51]. In addition, the hanger temperature can be influenced by the large overall surface area of the surrounding rock surface which is exposed to direct sunlight and will achieve a higher temperature than the air temperature. The potentially higher than air surface temperatures would be expected to contribute to the evaporative concentrating cycles necessary for halide salt-induced TGSCC in coldworked, austenitic stainless steels.

The salt composition found on the incident hanger surface was not consistent with sea salt which would be expected to contain more Na and less Mg, suggesting that Mg and Ca leach from the limestone and redeposit on the hanger. Mg and Ca salts appear to have assisted the Cl-induced TGSCC observed by making the halide films more concentrated.

Conclusion

- Ambient temperature, Cl-induced TGSCC was found to be the failure mechanism responsible for the failure of the cold-worked, austenitic stainless steel climbing anchors used on tower karst formations in southern Thailand.
- The Cl-induced TGSCC appeared to be assisted by acidic soil waters derived from the tropical vegetation. The acidic waters are associated with the high limestone dissolution–reprecipitation rates responsible for the tower karst formation. The steep, overhanging seaside rock formations may allow for high concentrations of Cl salts to accumulate including MgCl₂.
- Although the RH level in the region frequently reaches 100%, the average monthly low RH levels reported for the Phuket, Thailand area are often between 50 and 60% particularly in the winter. Daily low RH levels below 50% are not uncommon. This RH level is similar to indoor swimming pool atmospheres (50–60%) and is apparently within the range necessary for ambient temperature TGSCC to occur, i.e., between 25 and 45%, as previously determined from prior research on stainless steel contaminated with MgCl₂.
- Based on boiling MgCl₂ tests, the unalloyed welded cold-shut and Ti-6Al-4V U-bolts appear to be resistant to Cl-induced TGSCC and are suitable for use in this environment.

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