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# The Chloride Stress-Corrosion Cracking Behavior of Stainless Steels under Different Test Methods

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**Chloride-induced stress-corrosion cracking (SCC) is one of the failure modes of stainless steels. Highly alloyed austenitic stainless steels S32654, S31254, and N08028, and duplex grades S32750 and S31803 possess much improved resistance to SCC compared with S30400 and S31600 steels. With the development of a database, SSData, experimental data collected from calcium chloride tests, autoclave tests, and drop evaporation tests were evaluated. Stress-corrosion cracking data generated by autoclave tests agreed well with the practical service conditions and can be used to discriminate alloys for SCC resistance in sodium chloride solution. Drop evaporation test data can be used in situations where evaporation may occur and cyclic loading may be involved. The SCC resistance of alloys under each method increased with increasing molybdenum equivalent Mo + 0.25Cr + 0.1Ni. For a given alloy, the testing result depends on the stress state and environment; different test methods can give different ranking orders concerning SCC resistance. The performance of duplex stainless steels in a chloride-containing environment at higher temperatures was not as good as expected when dynamic loading was involved.**

Keywords

autoclave test, calcium chloride test, chloride stress-corrosion cracking, constant load test, drop evaporation test, magnesium chloride test, stainless steel

## 1. Introduction

STAINLESS steels have achieved extensive applications in food, pulp, paper, chemical, and petrochemical industries due to their good resistance to general corrosion. However, austenitic stainless steels, which are characterized as tough and highly ductile in most environments, could fail in an essentially brittle manner at relatively low stresses or stress intensity factors when exposed to hot chloride solutions. The brittle fracture caused by the simultaneous presence of tensile stress and a specific corrosive medium is the so-called stress-corrosion cracking. The consequence of SCC is serious since it can occur at stress well below the typical range of design stresses, deteriorating both the reliability and the safety of equipment. Even though it was invoked that most alloys will sustain SCC in most environments if neglecting the kinetic aspect of SCC (Ref 1), the tendency towards SCC differs between alloys. For example, Fe-Cr-Ni alloys containing 30% chromium and 60% nickel could actually be immune to SCC in chloride solutions (Ref 2). Previously, designers had access to only a few materials that were intermediate between the stainless steels, such as S31703 and N08904, and the nickel-base alloys, such as N06625 and N10276. This situation has been ameliorated by the introduction of highly alloyed austenitic and duplex stainless steels. These steels possess much better SCC resistance than conventional S30400 and S31600 austenitic grades. A distinguishing characteristic of these highly alloyed stainless steels is that they can be delivered at a much lower price than nickel-base alloys. With progress in the development of new stainless steels, reliable data on various types of properties obviously must be available in order to choose materials suitable for a given application (Ref 3). If data cannot be found for a given material, it often will be disregarded in the process of materials selection. The need for material data is recognized for materials selection, which is a basic domain of computer aided design, and for other applied science and technologies (Ref 4).

This paper compares the chloride SCC behavior of highly alloyed austenitic and duplex stainless steels in calcium chloride, autoclave, and drop evaporation tests and presents the results from the assessment of testing data for these alloys.

## 2. General Conditions of Inducing Stress-Corrosion Cracking

The first case of SCC in stainless steels was reported in the mid-1930s (Ref 5). The essential variables affecting SCC consist of an aggressive environment, state of stress, alloy composition and microstructure. The critical stress state is dependent on alloy and environment; therefore, these three variables interact.

Stress-corrosion cracking occurs in water containing less than 10 ppm chloride, fluoride, sulfate, polythionate ions, and in NaOH, KOH solutions, etc., but most cases of SCC for stainless steels probably occur in chloride ion solutions. Besides the influences of the particular anions, temperature plays an important role in SCC. Tests in simulated occluded cells reveal that SCC is much easier to develop and propagate at 90 °C than at 50 °C (Ref 6). Exposure tests indicate that neutral chloride ion solutions will not induce SCC unless the temperature exceeds 60 °C, although fluoride, polythionate, and acidified chloride solutions were reported to induce SCC in sensitized austenitic stainless steels even at ambient temperatures (Ref 7-9). Stress-corrosion cracking in NaOH and KOH solutions is not observed unless the temperature exceeds at least 60 °C, and SCC of sensitized stainless steels in high-purity water with oxygen concentration higher than 0.2 ppm requires temperatures above 120 °C. In addition to the effects of particular anions and tem-

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perature, the existence of tensile stress, either applied or residual, is considered as a prerequisite for SCC to occur.

## 3. Testing Methods

Stress-corrosion cracking data were collected from Swedish laboratories. Three different types of constant load tests were used: calcium chloride tests, autoclave tests, and drop evaporation tests. All testing materials were solution treated.

### 3.1 *Calcium Chloride Tests*

The boiling magnesium chloride test, standardized as ASTM G36, was the first one introduced to investigate the SCC susceptibility of austenitic stainless steels and nickel-base alloys. This standard specifies that the test solution should be maintained at a constant boiling temperature of 155 °C, which corresponds to a concentration of about 45%. Due to historical reasons, extensive data were published on the use of boiling 42% magnesium chloride solution. However, the boiling magnesium chloride test involves a very severe environment. The qualitative ranking of austenitic stainless steels with respect to SCC resistance in this solution disagrees with observations in more dilute chloride solutions (Ref 10-12). Because of drawbacks of the magnesium chloride solution, a quasi-neutral (pH 6.5) calcium chloride solution was used (Ref 10-12). The test was performed in an aerated 40% calcium chloride solution at 100 °C with a constant load applied to the tensile specimen. The applied load was varied to enable the determination of the threshold stress, which is defined as the lowest stress below which no SCC failure will occur in a 500 h testing period.

### 3.2 *Autoclave Tests*

Autoclave tests (Ref 12) were performed in a chloride ion solution with an oxygen content of approximately 8 ppm and under the pressure of 100 bar. The specimen was spring loaded in tension, and a 0.2% yield stress was applied. Six specimens were simultaneously tested. If one of these specimens cracked, the alloy was considered susceptible to SCC. With variations in temperatures and chloride ion concentrations, a series of data points ($T$, $C_{Cl}$), below which no SCC will occur in a 1000 h testing period, can be determined.

### 3.3 *Drop Evaporation Tests (DET)*

The drop evaporation test (Ref 13, 14) was developed at Studsvik Energiteknik AB, Sweden, and later implemented as method MTI-5 in the MTI manual (Ref 15). The purpose of the test was to determine the relative resistance of iron- and nickel-base alloys to SCC in a sodium chloride drop evaporation system, due to the recognition that the initial dilute and thereby harmless chloride solution can turn into a concentrated one. This condensed electrolyte with a minor volume generates a relatively small risk for perforation of pits and crevice corrosion, but it might induce a great risk of SCC under evaporative conditions because the crack propagation rate of SCC can be many times the propagation rate of pits. In the test, the specimen was attached horizontally to two holders, and the load was applied vertically by weights connected to the holders by steel wires over rollers. The uniaxial load was applied as a fraction of the 0.2% yield strength of the alloy at 200 °C. Immediately after loading, the specimen was resistance heated to 300 °C with a current of 20 to 21 A and a voltage of about 0.3 V across the specimen. Aerated 0.1 M sodium chloride solution is then dripped onto the specimen at a rate of 6 drops/min ($f = 0.1$ Hz).

**Table 1 Results of stress corrosion tests and minimum yield stresses**

| UNS number | Material composition | Designation | $CaCl_2$ test(a), % | DET(b), % | Critical temperature at 0.05% $Cl^-$ (c), °C | Minimum 0.2% yield strength, MPa |
|---|---|---|---|---|---|---|
| **Austenitic stainless steels** | | | | | | |
| S32654 | 24Cr-22Ni-7Mo-0.5N | 654 SMO™ | >90 | >100 | >300 | 430 |
| S31254 | 20Cr-18Ni-6Mo-0.2N | 254 SMO™ | >90 | ≥90 | >300 | 300 |
| N08028 | 27Cr-31Ni-3.5Mo-1Cu | Sanicro 28 | 90 | ... | 230 | 220 |
| N08904 | 20Cr-25Ni-4.5Mo-1.5Cu | 904L | 90 | ≥70 | 183 | 220 |
| N08825 | 22Cr-40Ni-3Mo-2Cu | Sanicro 41 | 90 | ... | ... | 240 |
| S31050 | 25Cr-22Ni-2Mo | 2RE69 | 38 | ... | ... | 270 |
| S31603 | 17Cr-12Ni-2.5Mo | 3R60 | 35 | ≤10 | ... | 220 |
| S31000 | 25Cr-20Ni | 2RE10 | 20 | ... | 52 | 210 |
| S30403 | 18Cr-9Ni | 3R12 | 12 | ≤10 | 52 | 210 |
| **Duplex stainless steels** | | | | | | |
| S32750 | 25Cr-7Ni-4Mo-0.3N | SAF 2057® | 90 | ≥70 | >300 | 550 |
| S31803 | 22Cr-5Ni-3Mo-0.15N | SAF 2205® | 90 | ≥40 | 183 | 450 |
| S31500 | 19Cr-5Ni-2.5Mo-0.1N | 3RE60 | 90 | ... | 170 | 450 |
| S32900 | 26Cr-5Ni-1.5Mo | 10RE51 | 70(d) | ... | ... | 485 |
| S32304 | 23Cr-4Ni-0.1N | SAF 2304® | 85 | ≥40 | 138 | 400 |

(a) Ratio between threshold stress and UTS. Time to failure is 500 h unless specified (Ref 12). (b) Ratio between threshold stress and 0.2% yield strength ($R_{P_{0.2}}$) in drop evaporation tests (Ref 14). (c) Critical temperature at 0.05% chloride ion concentration in autoclave tests. (d) Time to failure is greater than 1000 h.

The test was continued until the specimen fractured, with a maximum time of 500 h.

## 4. Testing Results

### 4.1 *Constant Load Tests in Calcium Chloride Solution*

The calcium chloride testing results (Ref 12) are presented in Table 1 as a ratio between the threshold stress and tensile strength. The calcium chloride test can distinguish well the relative SCC resistance between standard grades, such as S31603 and S30403, but it ranks highly alloyed austenitic and duplex stainless steels at the same level. It suggests that a 500 h testing period is not enough to develop SCC for highly alloyed stainless steels, and a longer testing time should be used. This is the main drawback of the calcium chloride test, particularly for testing highly alloyed stainless steels. The testing results show that molybdenum bearing austenitic alloy S31050 exhibits a slightly better SCC resistance than S31000. However, the positive effect of molybdenum will remain only if the chloride solution is in a neutral state. As discussed later in this paper, molybdenum gives a negative effect on SCC resistance in acidified calcium chloride solution. Figure 1 shows that the applied load in percentage of ultimate tensile strength (UTS) increases with increasing molybdenum equivalent Mo + 0.25Cr + 0.1Ni, particularly for the austenitic grades. The deviation of a data point corresponding to N08825 may be attributed to its very higher nickel content in the alloy because sufficient nickel contents in austenitic grades enhance SCC resistance.

### 4.2 *Autoclave Tests in Chloride Ion Solutions*

The results of autoclave tests are presented as a function of critical temperature and chloride ion concentration (Ref 12), as shown in Fig. 2. An example of the determination of critical temperature for N08028 is illustrated. The critical temperatures at 0.05% chloride ion concentration for these alloys are also presented in Table 1. The figure shows that there exists a well-defined relationship between critical temperature, $T$, and chloride ion concentration, $C_{Cl}$. For each given ($T_0$, $C_0$) and alloy, if $T_0 \leq T$ and $C_0 < C_{Cl}$, then the alloy can withstand the environment attack without showing SCC within a testing period of 1000 h. Autoclave tests clearly identify the relative SCC resistance among N08028, N08904, S31803, and S31500. Super austenitic steel S31254 and duplex alloy S32750 possess good SCC resistance, and no SCC is observed within a 1000 h testing period. Austenitic alloy N08028 is resistant to SCC in aerated solutions with chloride ion concentrations up to 100 ppm and oxygen 8 ppm at 250 °C and above, whereas alloy N08825 shows heavy cracking (Ref 16). Referring to SCC resistance, duplex steel S32750 is superior to austenitic grade N08028, and S31803 is better than N08904. These results confirm the observations that duplex stainless steels possess better SCC resistance than austenitic grades with a comparable chromium content. The improved resistance to SCC for duplex stainless steels has been attributed to the effect of ferrite in the austenitic matrix on blocking the progress of cracks (Ref 17). With a similar type of duplex microstructure, the positive effect of molybdenum on enhancing SCC resistance is clearly identified by S31803 because Cr-Ni type duplex grade S32304 possesses about the same contents of chromium and nickel as Cr-Ni-Mo duplex steel S31803. For both duplex and austenitic stainless steels, the critical temperature increases with increasing molybdenum equivalent Mo + 0.25Cr + 0.1Ni, as shown in Fig. 3. Figure 3 also indicates that the molybdenum contributes more to duplex grades than to austenitic ones with regard to SCC resistance.

### 4.3 *Drop Evaporation Tests*

The results of DET (Ref 14) are presented in Table 1 as a ratio between the threshold stress and 0.2% yield strength on the alloy. As indicated by the data, DET results rank the austenitic grades S32654 and S31254 with high SCC resistance, and the duplex steels S31803 and S32304 with relatively lower SCC resistance although the ranking is still above the conventional S30400 and S31600 steels. The microscopic examinations reveal that the morphologies of cracking are transgranular for both highly alloyed austenitic and duplex stainless steels, but they are less branched than the classical SCC. Selective dissolution of ferrite in duplex stainless steels was observed (Ref

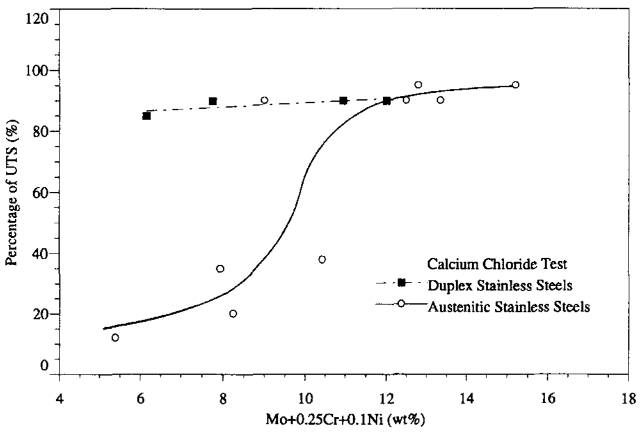

**Fig. 1** Dependence of threshold stress in percentage of UTS on molybdenum equivalent for austenitic and duplex stainless steels.

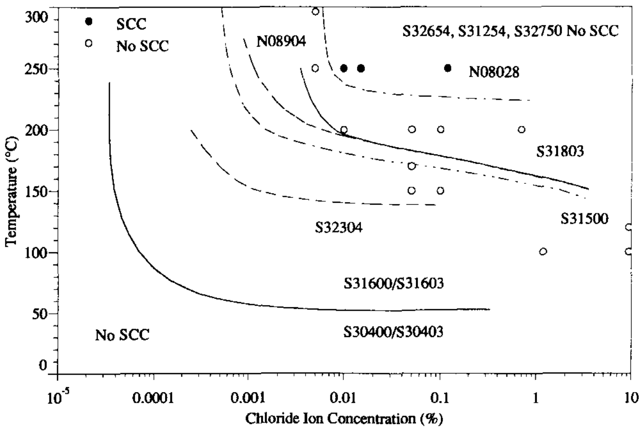

**Fig. 2** Results of autoclave tests in aerated neutral chloride ion solutions. Determination of critical temperature is illustrated for N08028.

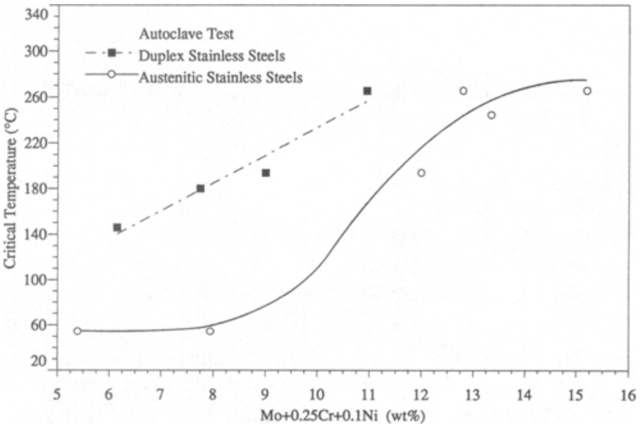

**Fig. 3** Dependence of critical temperature on molybdenum equivalent in 0.05% chloride ion solution for austenitic and duplex stainless steels.

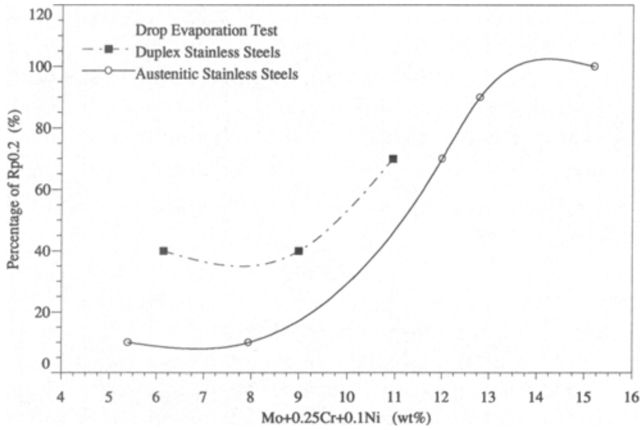

**Fig. 4** Dependence of threshold stress in percentage of 0.2% yield strength on molybdenum equivalent for austenitic and duplex stainless steels.

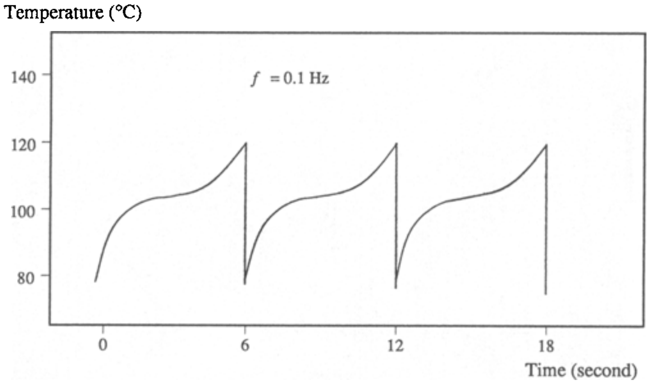

**Fig. 5** Temperature profile on specimen surface in drop evaporation test.

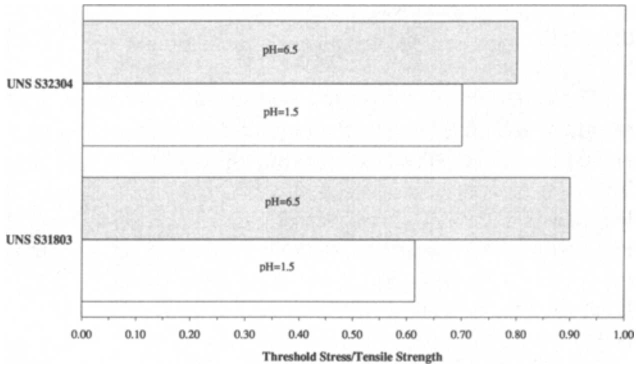

**Fig. 6** Effect of pH value on the relative order of threshold stress to tensile strength ratio for Cr-Ni-Mo and Cr-Ni duplex stainless steels in boiling $CaCl_2$ solution at 100 °C.

14). As in the calcium chloride test, Fig. 4 reveals that the allowable load in percentage of yield strength increases with the molybdenum equivalent Mo + 0.25Cr + 0.1Ni for both austenitic and duplex grades.

## 5. Discussion

### 5.1 *Influence of Stress State*

The testing results are dependent on the test methods used. For example, in autoclave tests S32750 has a similar SCC resistance to S31254, and S31803 is comparable to N08904 if chloride ion concentration is higher than 0.01%. However, both duplex steels exhibit a SCC resistance lower than the corresponding austenitic grades in DET. Similar results were reported when duplex steel S32900 was compared with S31600 (Ref 13). The change in the relative order of SCC resistance in DET might be associated with the stress state that differs from the one used in the autoclave test. When the specimen surface is wetted and dried with a frequency of 0.1 Hz in DET, the temperature on the specimen surface varies with time due to the evaporation of drops, as illustrated in Fig. 5. This variation may induce residual stresses on the surface and result in thermal fatigue, facilitating occlusion formation that is necessary to inhibit repassivation (Ref 18).

The dependence of testing results on stress state can also be seen in the cyclic slow strain rate tests performed in aerated 50% lithium chloride solution at 100 °C. Both S32304 and S31083 fail after cycling 5 times in tests (Ref 19), and S32750 performs no better than the former grades (Ref 20), whereas failure occurs in S31254 after cycling 12 times (Ref 19). Therefore, highly alloyed austenitic grades possess better SCC resistance than duplex grades in the dynamic loading condition.

### 5.2 *Effects of Alloy Composition and pH Value*

As indicated by the autoclave tests and calcium chloride tests, molybdenum plays an important role in enhancing the SCC resistance in the neutral chloride ion solutions. However, the beneficial effect of molybdenum is known to vary with the pH value of the solution. Calcium chloride tests reveal that the ratio of threshold stress to tensile strength for Cr-Ni-Mo duplex steel S31803 is lower than Cr-Ni duplex grade S32304 at pH 1.5 in contrast to the relative order at pH 6.5 (Ref 12), as shown

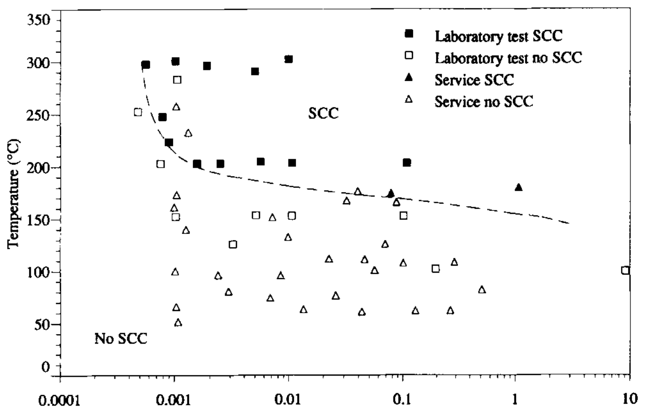

**Fig. 7** Comparison between autoclave test results and service experience for S31500. From Ref 12.

in Fig. 6. This result strongly resembles the performance of these alloys in boiling magnesium chloride solution where S31803 performed worse than S32304.

The effect of nickel on the SCC resistance of austenitic stainless steels has been well recognized (Ref 21-23). With increasing nickel content, the stacking fault energy is enhanced. This will in turn prompt cross-slip and make the slip process less planar, thereby reducing stress concentrations at the tip of blocked slip band and mitigating the possibility of the rupture of surface film. This principle is realized by alloys S32654, N08028, and S31254, each of which contains 20% or more Ni. However, it can be applied only to steels with an austenitic microstructure because the addition of a small amount of nickel to ferritic stainless steel induces the susceptibility to SCC (Ref 24).

### 5.3 *Applicability of Testing Results*

All of the accelerated tests discussed above are useful for accumulating valuable information in a short time but may not provide accurate data with design significance because applications of higher stress, higher temperature, and higher concentration of aggressive solutions may not simulate the situations in service. However, data collected from field tests for S31500 indicate that autoclave tests can mirror more closely a practical service condition than magnesium chloride or calcium chloride tests (Ref 12), as shown in Fig. 7. Since most aqueous media are in practice based on aerated sodium chloride solutions, the results from autoclave tests can be used to discriminate alloys for their resistance to SCC. Drop evaporation test data are suitable for use in chloride-containing environments in which cyclic stresses may be involved, particularly at elevated temperatures.

The data obtained with accelerated tests cannot be used directly for design purpose. However, the higher stresses that can be used for highly alloyed austenitic and duplex stainless steels indicate that a design engineer upgrading from S30400 and S31600 grades to those highly alloyed stainless steels has the opportunity for construction economies by down gage or by increasing load when possible SCC is involved.

## 6. Conclusions

- The relative resistance of highly alloyed austenitic stainless steels and duplex stainless steels to SCC in calcium chloride tests, autoclave tests, and drop evaporation tests is identified. The SCC resistance of alloys measured by three different methods increased with increasing molybdenum equivalent Mo + 0.25Cr + 0.1Ni in neutral chloride ion solutions. The performance of duplex stainless steels in a chloride-containing environment at the higher temperatures was not as good as expected when dynamic loading was involved.
- Stress-corrosion cracking data obtained from autoclave tests agree well with the actual service conditions. Drop evaporation test results can be used to estimate the SCC susceptibility of stainless steels in applications where evaporation may occur and cyclic loading may be involved. Since calcium chloride is milder than magnesium chloride solution, the time to fracture in calcium chloride tests for highly alloyed stainless steel is too long to be practical for laboratory testing.
- The conditions for SCC to occur depend on the stress state, environment, and alloy. Therefore, different test methods can give different ranking orders with respect to SCC resistance.

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