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# Evaluation of stress corrosion cracking susceptibility of stainless steel 304L with surface nanocrystallization by small punch test

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ABSTRACT

In this study, the small punch test (SPT) was conducted to evaluate the stress corrosion cracking (SCC) susceptibility of stainless steel (SS) 304L with surface nanocrystallization (SNC) in 1 mol/L NaCl+0.5 mol/L HCl aq. The surface mechanical attrition treatment (SMAT) was applied to realize the SNC. The mechanical property and micro-structural evolutions of SS 304L induced by SMAT were investigated through optical microscope (OM), X-ray diffraction (XRD), micro-Vickers hardness and transmission electron microscopy (TEM). The grain size on the surface of the material was reduced to 30–100 nm. The SPT was conducted in both ambient air and corrosive solution. The results were investigated by OM and scanning electron microscopy (SEM), showing that in ambient air, the specimen with 30 min SMAT performed a higher yield strength and lower ductility than the solution annealed (SA) counterpart. The SS 304L without SMAT presented a transgranular SCC (TGSCC) mode in chloride solution. In contrast, the SNC 304L SS showed a higher SCC susceptibility with a typical intergranular SCC (IGSCC).



## 1. Introduction

Recently the surface nanocrystallization (SNC) of metallic material has been brought into focus. Generally, there are three techniques to generate nano-grains on the surface of the metallic material: surface coating (or deposition), self-nanocrystallization and compounding nanocrystallization. The self-nanocrystallization technique has been studied intensively due to the firm nano-grain produced by this method and no obvious interface with the metallic matrix. This method was applied to improve the surface free energy by using a nonequilibrium method to produce nano-grains on the surface of material. Many modified techniques have been developed for self-nanocrystallization, such as surface mechanical attrition treatment (SMAT) [1], ultrasonic impact peening (UIP) [2], fine particle peening (FPP), laser-shock peening (LSP) [3], surface mechanical grinding treatment (SMGT) and etc.

SNC is a promising technique which combines the nano technique and conventional metallic material to produce a novel material with special structure and enhanced exterior property which has a enormous application potential. The previous researchers have studied the tensile strength, fatigue property [4], structural transformation [5], thermal stability [6] and anti-pitting or general corrosion property [1,7] of the SNC material. However, only a few researchers study the stress corrosion cracking (SCC) susceptibility of the materials with nano-grain surface. Shi et al. [8] used the constant load method to evaluate the SCC susceptibility of SS 316L with SMAT, they concluded that the SMATed samples' resistance to SCC susceptibility was enhanced, but the resistance to pitting was reduced. Ling et al. [9] immersed the SS 304 welded joints samples with UIP in the boiling 42% magnesium chloride solution to evaluate the effect of UIP on the SCC resistance, an improvement of the SCC resistance of the weld joints with UIP was observed. But we consider the materials' structure and property evolution during SNC process have negative effects on the materials' SCC resistance, all the previous researchers used the samples with both surface residual compressive stress and structure evolution produced during SNC process which could not reflect the sole effect of the structure and property evolution on the SCC resistance. The contribution of surface compressive stress to enhancement of SCC resistance was testified long ago, it is of significance to figure out whether the

*Abbreviations:* SPT, Small punch test; SCC, Stress corrosion cracking; SS, Stainless steel; SNC, Surface nanocrystallization; SMAT, Surface mechanical attrition treatment; OM, Optical microscope; XRD, X-ray diffraction; TEM, Transmission electron microscopy; SEM, Scanning electron microscopy; SA, Solution annealed; TGSCC, Transgranular stress corrosion cracking; IGSCC, Intergranular stress corrosion cracking; UIP, Ultrasonic impact peening; FPP, Fine particle peening; LSP, Laser-shock peening; SMGT, Surface mechanical grinding treatment; SSRT, Slow strain rate test; AE, Acoustic emission; Fcc, Face-centered cubic; ASS, Austenitic stainless steel; HIC, Hydrogen-induced cracking; HISCC, Hydrogen-induced stress corrosion cracking; HFAD, Hydrogen-facilitated anodic dissolution; CW, Cold work; HEE, Hydrogen environment embrittlement

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structure and property evolution during the SNC process has a positive effect on the materials' SCC susceptibility or not and its mechanism.

Constant strain, constant load, $K_{ISCC}$ and slow strain rate test (SSRT) are the common methods for evaluating the SCC behavior of metals. However, each of them has its own disadvantages more or less. The first three methods' defects are time-consuming, requiring lots of specimens and low reproducibility. Although SSRT could shorten the test period, it has a strict requirement with the specimen's size, i.e., it was not able to be applied to the small size samples or specimens with certain special treatment on single side. Therefore, there is a need for a novel SCC test method using miniaturized specimens which is able to avoid the above disadvantages. Small punch test (SPT) is a newly arisen technique which is widely used to evaluate the tensile strength, creep strength, fatigue property and other mechanical properties of the materials as a non-destructive technique which is unable to affect integrality of in-service equipment. It is scarce to evaluate the SCC susceptibility using SPT method according the published literatures. Yu et al. [10] evaluated the SCC property of the hot-rolled HT80 in synthetic sea water using SPT and acoustic emission (AE) which proved that the SPT is an useful method to evaluate the SCC susceptibility of high-strength steel.

In this study, we use SMAT to generate nano-grains on the stainless steel (SS) 304L surface, and then evaluate the SCC susceptibility of the material with SNC by using the SPT technique.

## 2. Experimental procedure

### 2.1. Material

The material used in this study is a commercial type SS 304L which is in the solution annealed (SA) condition. The chemical composition obtained by elemental analysis is 0.021%C, 18.04%Cr, 8.02%Ni, 0.345%Si, 1.64%Mn, 0.037%P, 0.001%S and Fe balance. The tensile strengths of this material are 605 MPa, 300 MPa of yield strength and the elongation is 64.5%.

### 2.2. SNC by SMAT

The SMAT method is applied to generate nano-grains on the SS 304L surface. The experimental parameters are vibration frequency 50 Hz, 8 mm diameter steel balls, processing time 30 min. Fig. 1 shows the schematic diagram of the SMAT device. Hardness test was conducted on a Micro–Vickers Hardness Tester with applied load 0.98 N and holding time 15 s. X-ray diffraction (XRD) was used to examine the martensite transformation. The micro-structural evolutions of the SS 304L after SMAT was investigated through optical microscopy (OM) and transmission electron microscopy (TEM).

### 2.3. SPT technique for evaluating SCC susceptibility

The effect of environmental parameters like pH value, chloride content, temperature and humidity on the SCC susceptibility have been critically examined. The study clearly delineated two necessary conditions for ambient temperature SCC: (a) pH value is at the range of $-0.5$–$0.5$ and (b) $Cl^-$ concentration is from 0.5 to 5 M [11].

Fig. 2 shows the schematic diagram of the SPT device. The upper and lower dies of the SPT jigs were made of SS 304L to prevent corrosion and the occurrence of a galvanic couple. The aperture $d_2$ on lower die is 4 mm and the angle of chamfer is $0.5 \times 45^\circ$. For conducting the SCC test, the whole SPT device was immersed in a tank which was made of SS 304L and filled with the corrosive solution. A through-hole in 10 mm diameter was made on the lower die ($d_3$) to enable the specimen immersed in the corrosive environment throughout. The size of the specimen was 10 mm in diameter and $0.5 \pm 0.01$ mm in thickness ($t_0$). The residual compressive stress on the material surface after SMAT was eliminated due to the destruction of the elastic–plastic structure during sampling the SPT specimens. Loaded onto the top surface of the specimen used a steel ball of 2.5 mm in diameter and hardness HRC > 55. The punch diameter ($d_1$) is also 2.5 mm.

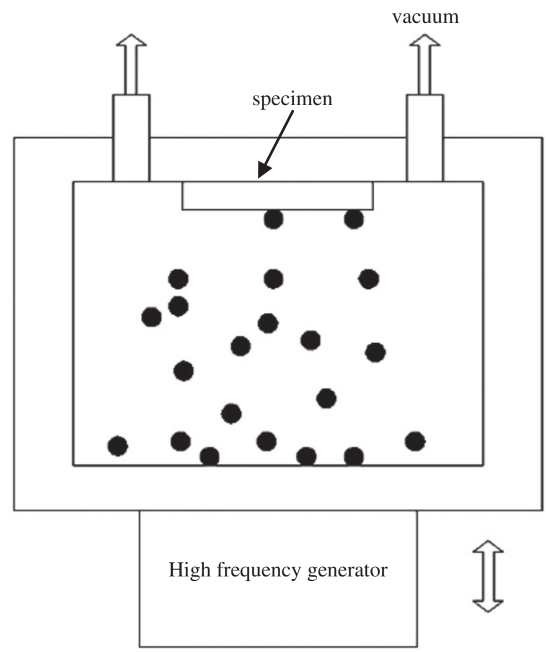


**Fig. 1.** Schematic diagram of the SMAT device.

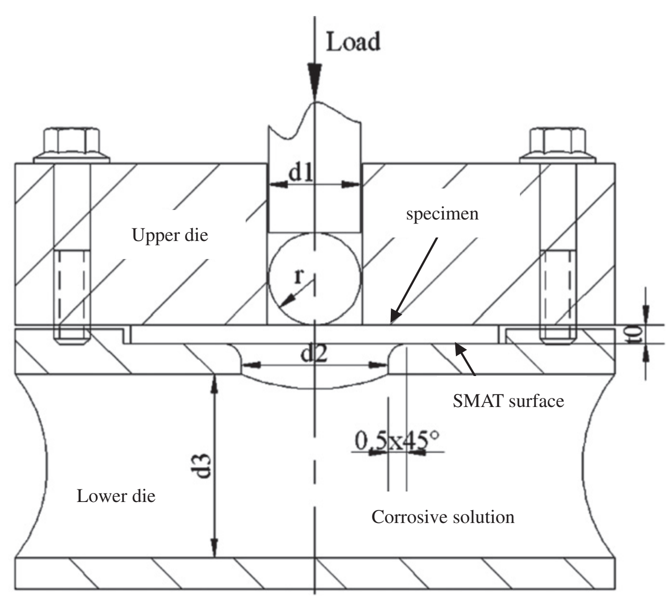


**Fig. 2.** Schematic diagram of SPT for evaluating SCC susceptibility.

The SPT was performed on a universal tensile testing machine. The experimental temperature was constant 25 °C. The loading rate was $3 \times 10^{-3}$ mm/min which was in the susceptive range of the SCC. The experimental data were collected with the frequency of 0.1 Hz. The 1 mol/L NaCl+0.5 mol/L HCl aq was used as a corrosive environment. For the SNC specimen, the SMAT surface