Salt-Fog Corrosion Behavior of GCr15 Steels Treated by Ultrasonic Strengthening Grinding Process

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Abstract: In this paper, the corrosion resistance of four GCr15 steel samples has been investigated. Three samples were initially surface-treated by ultrasonic shot peening (USP), a dry ultrasonic strengthening grinding process (DUSGP), and a wet ultrasonic strengthening grinding process (WUSGP), respectively, while another one was only polished. Then, all the processed samples were subjected to salt spray corrosion. Sample properties, such as capacitance arc, self-corrosion potential ($E_{corr}$), self-corrosion current density ($I_{corr}$), corrosion morphology, and corrosion products were analyzed. Results show that the sample treated by WUSGP has the best corrosion resistance, which can be attributed to the high dislocation density, small grain size, needle-like and lath-like shape of martensite content formed in the treatment, which hinders $Cl^-$ from eroding the matrix. The obtained corrosion pits and cracks in Sample WUSGP show a width of approximately 1.4 $\mu$m and 2.1 $\mu$m, respectively, degrading 78% and 75% compared to polishing. The possible corrosion mechanism of the samples has been discussed. The findings denote that the treatment fused ceramic balls, strengthened liquid, and corundum in USP could be an efficient method to improve the corrosion resistance of some mechanical parts.

Keywords: GCr15 steel; ultrasonic strengthening grinding process; salt spray corrosion; corrosion resistance

1. Introduction

GCr15 steels are widely used to manufacture mechanical components, such as bearings [1,2], ball screws [3], and steel balls [4], mainly due to their high hardness and wear resistance [3]. These mechanical components have played a significant role in many industrial fields, such as automotive, aviation, mining, and marine equipment [3,5]. Metals are easily corroded when working in a marine environment, which causes economic and material loss for marine equipment [6]. The corrosion of metal components can be attributed to multiple factors, including temperature [7,8], the PH value of the seawater [9,10], micro-organisms [11], etc. Consequently, a severe degradation occurs in the mechanical properties of the metals and finally leads to fatigue failure.

To address this issue, many methods have been proposed for the long-term protection of steel components, such as surface coating [12–15] and surface chemical heat treatment [16–18]. In particular, surface coating attracts widespread research interests, and great efforts for developing high-performance protective coating have been studied.
For example, Al coating developed by the thermal-sprayed method can provide efficient protection for steels due to the existence of passivating film [19,20]. Nevertheless, a serious pitting problem occurs once the passivating film has been damaged [21,22]. Moreover, the Al coatings formed by thermal-spraying are easy to peel off under heavy loads [23]. Zn-Al [12,24], Zr-Ti [13,25], and NiCr-Cr$_2$C$_2$ [14,15] coatings have also been studied broadly. An example of corrosion behavior of a Zn-Al composite coating in a salt spray corrosive environment, which exhibits the excellent anti-corrosion properties by absorbing the benefits of both Zn and Al, has been investigated by Zhao et al. [12]. However, the good corrosion resistance of Zn-Al coatings is only suitable for low-carbon steel substrates [12]. With the rapid development of the mechanical industry, new techniques to improve the anti-corrosion properties of steel are urgently needed.

As an alternative, mechanical strengthening has emerged as a promising solution to obtain a good corrosion resistance for metals, mainly due to the chilled layer and compressive residual stress microstructures formed on metal surfaces after surface treatment [26–28], which results in the improvement of fatigue life. So far, mechanical strengthening techniques, such as shot peening (SP) [29,30] and rolling finishing [31,32] have been proposed extensively for corrosion resistance investigation of metals. However, traditional shot peening typically produces a high roughness on the metal surface, and the protection ability is still far from the practical demands [33]. On the other hand, the hardened layer produced by rolling and the internal material have an obvious delamination phenomenon [34], which easily causes the surface layer to fall off. In contrast, ultrasonic shot peening has the characteristics of low cost, large shape correction range, small footprint, good controllability and repeatability. Surprisingly, the use of ultrasonic shot peening for improving the corrosion resistance of GCr15 steels is poorly investigated.

In this work, the corrosion behavior of four GCr15 steel samples has been investigated in a salt spray corrosive environment. Samples are surface-treated with ultrasonic shot peening (USP), a dry ultrasonic strengthening grinding process (DUSGP), a wet ultrasonic strengthening grinding process (WUSGP), and polishing, respectively. The capacitance arc and polarization curves of these samples were studied. The corrosion morphology and products were further analyzed to reveal possible corrosion mechanisms.

2. Materials and Methods

2.1. Samples Preparation

Four GCr15 steel samples with geometrical parameters of 100 mm × 75 mm × 10 mm (See Figure 1a) were employed in this study. Their main chemical composition is shown in Table 1, which was analyzed by Energy Dispersive Spectroscopy (EDS). The heat treatment procedure is shown Figure 1b. The samples were initially quenched at a temperature of 855 °C to obtain a good hardness, followed by an oil cooling process. Then, the samples were treated with a tempering process of 120 min at 160 °C to reduce the brittleness of the quenched workpiece. Finally, the sample surfaces were polished with 200# sandpaper to obtain a roughness < 0.2 µm and cleaned with alcohol through an ultrasonic bath.

Figure 1. (a) The geometric parameters of samples; (b) the heating process.
Table 1. Chemical element composition of GCr15 steels.

| Chemical Element | C    | Si    | Mn   | Cr   | P    | S    | Ni   | Cu   |
|------------------|------|-------|------|------|------|------|------|------|
| Content (wt%)    | 0.95-1.05 | 0.15-0.35 | 0.20-0.40 | 1.30-1.60 | 0.027 | 0.02 | 0.30 | 0.25 |

2.2. Surface Treatment of the Samples

Three samples were surface-treated by ultrasonic processing equipment, while another sample was without surface treatment for experimental comparison. The working principle of the ultrasonic equipment is reported in Figure 2. First, ultrasonic waves were produced by an electro-mechanical ultrasonic transducer and then applied to a workpiece. An ultrasonic transducer was utilized to energize an acoustically tuned resonator bar, causing it to vibrate. Finally, the energy generated from these impulses was imparted to treat the surface through the ceramic balls. The main difference between the surface treatment of the three samples was the employment of different treatment materials, as depicted in Figure 2b–d. The working parameters of ultrasonic processing is presented in Table 2. One sample was treated with ceramic balls, while the other two samples were treated with mixed materials. The surface treatment that only considered ceramic balls was named USP, and mixed white corundum and the ceramic balls was named DUSGP. The other one that mixed white corundum and ceramic ball, as well as strengthening liquid was named WUSGP [35]. The strengthening liquid was composed of extrusion additive (i.e., C_{10}H_{14}N_{2}Na_{2}O_{8}·2H_{2}O), triethanolamine (C_{6}H_{15}NO_{3}) and water. The samples surface-treated by USP, DUSGP, WUSGP, and polishing are named Sample USP, DUSGP, WUSGP, and polished, respectively.

![Figure 2](image_url)

Figure 2. (a) Schematic diagram of ultrasonic processing equipment; (b) ultrasonic shot peening treatment; (c) dry ultrasonic strengthening grinding process treatment; (d) wet ultrasonic strengthening grinding process treatment.

2.3. Salt Spray Process of the Samples

Each processed sample was cut into three plates with a diameter of 10 mm to calculate the mean values of experimental data. The salt frog test was performed using HT-YW-60 salt spray test machine and based on the standard ASTM B117. The working temperature was 35 (±2) °C, the precipitation rate of the salt spray was 1.5 mL/(cm²·h) with an air pressure of 96 kpa. PH value was set in the range of 6.5–7.2, and the total spray time was 96 h.
2.4. Characterization

The cut samples were mounted with epoxy and then used to make electrodes by welding a wire on their surface. The electrochemical impedance spectroscopic (EIS) measurement has been carried out using Solartron 1260 + 1287 electrochemical workstation. The potentiodynamic polarization test was performed at a scan rate of 1.5 mV/s in the range of −0.5 V–5 V.

An X-ray diffractometer (XRD, Rigaku + UltimaIV, Rigaku Corp, Akishima, Tokyo, Japan) was used to analyze the phase and corrosion components of the samples. The scanning electron microscope (SEM, ZEISS Gemini 300, Carl Zeiss AG, Oberkochen, Germany) coupled with an energy dispersive spectrometer (EDS, Oxford X-Max 50, Oxford, UK) was employed to observe the surface micromorphology. Autofocus Microscope (GP−420H, Kunshan gaopin precision instrument co. LTD, Kunshan, China) was utilized to observe the macromorphology.

3. Results and Discussion

Figure 3 reports the Nyquist diagram of all the samples immersed in 3.5% NaCl solution made in the laboratory for 96 h. The capacitive loop was utilized to characterize the charge transfer resistance at the interface of corrosion products. It can be seen that the capacitive loop of Sample polished is much smaller than the other samples, which can be attributed to the accumulation of corrosion products. The capacitive loops of the samples with surface treatment are significantly improved. In particular, the biggest capacitive arc radius was obtained in Sample WUSGP. The magnitudes of the charge transfer resistance of all the samples are ranked as follows: WUSGP > DUSGP > USP > Polished.

![Figure 3. Nyquist plots of the samples immersed in seawater for 96 h.](image)

The corrosion potential ($E_{corr}$) and the corrosion current density ($I_{corr}$) are critical parameters to evaluate the performance of corrosion resistance, which are calculated through Tafel extrapolation [36]. The polarization curves of the samples and the obtained results are reported in Figure 4 and Table 3. It can be found that the Sample WUSGP has the largest corrosion potential (i.e., −0.3999), while the corrosion potentials of Sample DUSGP and USP are relatively close, realizing a value of −0.5926 V and −0.5122 V, respectively.

| Parameters                     | Value  |
|--------------------------------|--------|
| Vibration frequency (kHz)      | 20     |
| Peening distance (mm)          | 30     |
| Processing time (min)          | 3      |
| Diameter of ceramic balls (mm) | 1      |
| Grain size of brown corundum (µm)| 15     |

Table 2. Working parameters of ultrasonic processing.
This can be by explained considering the effect of the strengthening liquid in surface treatment, which degrades the corrosion potential of Sample WUSGP. Generally, the lower the corrosion potential, the worse the corrosion resistance. Furthermore, the corrosion current of sample WUSGP is two times lower than sample Polished, exhibiting a value of 9.435 mA/cm². A smaller corrosion current means a lower corrosion rate. Therefore, sample WUSGP possesses the best properties of corrosion resistance.

![Figure 4](image_url)

**Figure 4.** The polarization curves of all the samples.

**Table 3.** Corrosion potential ($E_{corr}$) and the corrosion current density ($I_{corr}$) obtained in electrochemical corrosion test for each sample.

| Serial Number | $E_{corr}$/V | $I_{corr}$/mA cm⁻² |
|---------------|-------------|-------------------|
| Polished      | -0.7309     | 18.15             |
| USP           | -0.5926     | 15.28             |
| DUSGP         | -0.5122     | 12.79             |
| WUSGP         | -0.3999     | 9.435             |

The X-ray diffraction patterns of all the samples are shown in Figure 5a. It is noted that the new diffraction peaks occurred in the surface-treated samples, such as (200). The new diffraction peak is particularly obvious in Sample WUSGP. To further understand the possible mechanism, the grain size and dislocation density of these samples have been calculated (See Figure 5b) through Williamson–Hall equations [37]. Sample polished has the maximum grain size and the minimal dislocation density, realizing a value of 28.7 µm and 0.3658 × 10¹⁶ m⁻², respectively. After surface treatment, the grain size decreases, and the dislocation density increases. In sample WUSGP, the grain size is refined to 6.45 µm, and the dislocation density increased to 2.639 × 10¹⁶ m⁻², which is mainly due to the plastic deformation of the surface tissue after intensive grinding and the phase transforms, resulting in an increase in the martensite content (See Figure 5a). The martensite structures typically show a shape of needle and lath, making the surface structure denser. Previous studies demonstrated that the dense structure of high dislocation density and small grain size can significantly improve the corrosion resistance of the material [38]. This evidence further illustrates the benefits that USGP may bring.
The width of corrosion cracks is larger than in other samples. The corroded area of Sample USP, morphology of Sample DUSGP; (Figure 6).

Figure 6 shows the surface macro-morphology of the samples. It is found that most regions on the matrix surface of Sample polished are corroded (See Figure 6a), and the width of corrosion cracks is larger than in other samples. The corroded area of Sample USP, DUSGP, and WUSGP is relatively smaller (See Figure 6b–d). Especially in Sample WUSGP, the uncorroded area occupied approximately half of the whole matrix. The white substance is CaCO$_3$ formed in the corrosion process because the OH$^-$ produced by cathodic reacts with HCO$_3^-$ and generates the CO$_3^{2-}$, which further reacted with the Ca on the sample surface [39]. Corrosion products were further analyzed by XRD, as depicted in Figure 7. In Sample polished, the corrosion products mainly consist of Fe, Fe$_2$O$_3$, Fe(OH)$_3$, and Fe$_3$O$_4$ (See Figure 7a), but in other samples, FeOOH appeared (See Figure 7b–d). This could be attributed to the chemical reaction between Fe(OH)$_2$ and O$_2$. In the initial phase of corrosion, the passive film Fe(OH)$_2$ on the sample surface will undergo a redox reaction with O$_2$ to form FeOOH. Over time, Fe(OH)$_2$ will absorb O$_2$ to produce reddish-brown Fe(OH)$_3$. At the same time, Cl$^-$ will intrude into the vulnerable regions of the matrix, resulting in the further oxygen absorption reaction of Fe(OH)$_3$ to form the black Fe$_3$O$_4$. It is also noted that the diffraction peak intensity of Fe is the strongest in Sample WUSGP, indicating that there are fewer corrosion areas, and the content of the formed FeOOH and Fe$_3$O$_4$ is relatively low.

Figure 6. Macroscopic corrosion morphology of each sample: (a) the corrosion macro-morphology of Sample polished; (b) the corrosion macro-morphology of Sample USP; (c) the corrosion macro-morphology of Sample DUSGP; (d) the corrosion macro-morphology of Sample WUSGP.
Figure 6. Macroscopic corrosion morphology of each sample: (a) the corrosion macro-morphology of Sample polished; (b) the corrosion macro-morphology of Sample USP; (c) the corrosion macro-morphology of Sample DUSGP; (d) the corrosion macro-morphology of Sample WUSGP.

Figure 7. XRD patterns of corrosion products for each sample: (a) the corrosion macro-morphology of Sample polished; (b) the corrosion macro-morphology of Sample USP; (c) the corrosion macro-morphology of Sample DUSGP; (d) the corrosion macro-morphology of Sample WUSGP.

The corrosion micromorphology of Sample polished, USP, DUSGP, and WUSGP are shown in Figure 8a–d, respectively. Corrosion holes in Sample polished have a diameter of approximately 6.4 μm, while sample WUSGP has a diameter of only approximately 1.4 μm, which degrades 78%. This can be explained by the surface plastic deformation of WUSGP-processed samples, resulting in grain refinement and a denser surface layer. Corrosion holes in these samples exhibit different shapes, such as island, cotton, and lath-like. This is because Cl\(^-\) gradually erodes the matrix’s weak regions during the corrosion process, forming countless corrosion micro-batteries and reacting with the corrosion solution. The internal stress of the matrix material and the accumulation of reaction energy push corrosion micro-bubbles to rupture, form “island-like” and “cotton-like” structures, and then further form pitting holes. As the corrosion process intensifies, the initial local pitting gradually develops into corrosion holes, which then connect with each other and finally form the cracks. Figure 9a–d report the micromorphology of corrosion cracks for Sample polished, USP, DUSGP, and WUSGP, respectively. Different widths of corrosion cracks are observed. The polished sample shows a width of approximately 8.4 μm, while samples that experienced surface treatment have a relatively narrow width. For instance, Sample WUSGP realizes a crack width of only 2.1 μm, which degrades 75% compared to the polishing.

The energy spectrum of the corrosion crack area has been analyzed using EDS. The element distribution and atomic percentage of the samples are shown in Figure 10 and Table 4, respectively. It can be found that the Fe atomic percentage gradually decreases after surface treatment, and a lowest percentage was obtained in Sample WUSGP. This is mainly due to the peeling of the rust layer as the thickness increases. The higher the Fe atomic percentage, the more intense the oxidation reaction and the wider the corrosion crack, which is consistent with the corrosion crack micromorphology analysis. In addition, there are fewer Cl, Ca, and other elements in the corrosion products, which are mainly produced in the deposition process of salt spraying. O atomic percentage increases after surface treatment, owing to the oxidation during the corrosion process.
of Cl elements, it can be seen that the percentage of Cl in Sample WUSGP is much higher than in other samples, indicating that the corrosion rust layer in this sample has strong adsorption on Cl\(^-\). This behavior makes it difficult for Cl\(^-\) to react with other substances. Thus, the corrosion resistance of Sample WUSGP has been significantly improved.

![Micro-morphology of corrosion holes for each sample](image1)

**Figure 8.** Micro-morphology of corrosion holes for each sample; (a) the micromorphology of corrosion holes for Sample polished; (b) the micromorphology of corrosion holes for Sample USP; (c) the micromorphology of corrosion holes for Sample DUSGP; (d) the micromorphology of corrosion holes for Sample WUSGP.

![Corrosion crack morphology of each sample](image2)

**Figure 9.** Corrosion crack morphology of each sample; (a) the corrosion crack morphology of Sample polished; (b) the corrosion crack morphology of Sample USP; (c) the corrosion crack morphology of Sample DUSGP; (d) the corrosion crack morphology of Sample WUSGP.
Figure 10. (a1,a2) are the surface element distribution and content of Sample polished, respectively; (b1,b2) are the surface element distribution and content of Sample USP, respectively; (c1,c2) are the surface element distribution and content of Sample DUSGP, respectively; (d1,d2) are the surface element distribution and content of Sample WUSGP, respectively.
which then reacts with OH− to form Fe(OH)2. However, Fe(OH)2 is unstable in the initial stage of corrosion, which will be decomposed into FeO or oxidized in the test chamber, resulting in a redox reaction to form FeOOH [40,41]. FeOOH is an effective oxidant (except oxygen), α-FeOOH has a relative high stability, while γ-FeOOH has a low stability, which results in the transfer of electrons during the corrosion process, pushing it to react with Fe2+ to generate Fe3O4.

\[
\begin{align*}
O_2 + 2H_2O + 4e^- &\rightarrow 4OH^- \\
Fe &\rightarrow Fe^{2+} + 2e^- \\
Fe^{2+} + 2Cl^- + 4H_2O &\rightarrow FeCl_2 + 4H_2O \\
FeCl_2 + 4H_2O &\rightarrow Fe(OH)_2 + 2Cl^- + 2H^+ + 2H_2O \\
Fe^{2+} + 2OH^- &\rightarrow Fe(OH)_2 \\
4Fe(OH)_2 + O_2 &\rightarrow 4FeOOH + 2H_2O \\
3\gamma - FeOOH + H^+ + e^- &\rightarrow Fe_3O_4 + 2H_2O \\
Fe^{2+} + 2\gamma - FeOOH &\rightarrow Fe_3O_4 + 2H^+ \\
6Fe(OH)_2 + O_2 &\rightarrow 2Fe_3O_4 + 6H_2O
\end{align*}
\]

The main corrosion mechanisms are described in Figure 11, and the details are depicted by the following chemical reaction equations (See Equation (1)). First, Na+ and Cl− are gradually accumulated on the sample surface, ferrite and cementite form numerous corrosion micro-batteries, and ferrite in the anode area preferentially dissolves to form Fe2+, which then reacts with OH− to form Fe(OH)2. However, Fe(OH)2 is unstable in the initial stage of corrosion, which will be decomposed into FeO or oxidized in the test chamber, resulting in a redox reaction to form FeOOH [40,41]. FeOOH is an effective oxidant (except oxygen), α-FeOOH has a relative high stability, while γ-FeOOH has a low stability, which results in the transfer of electrons during the corrosion process, pushing it to react with Fe2+ to generate Fe3O4.

Due to the presence of H+ in the corrosion solution, the chemical reaction between the dissolution of the corrosion product film Fe(OH)2 and the metal matrix material will be accelerated. However, the corrosion product film will also be eroded in redox reactions because Cl− is extremely corrosive, penetrating the corrosion product film to further accelerate corrosion, and the corrosion product gradually accumulates and forms a layer on
the sample surface. The thickness of the corrosion layer becomes thicker over time, which reduces the corrosion rate of Cl\(^{-}\) to the matrix material in the later stage of corrosion. In Sample USGP, a dense strengthening layer and film are formed on the surface after surface treatment. Thus, the corrosion rate of Cl\(^{-}\) was reduced in the initial stage of corrosion. The corrosion mainly occurs in the weak regions of the substrate and in the active anode area will trigger a redox reaction to produce Fe(OH)\(_2\) film. With the continuous erosion of Cl\(^{-}\), the migration rate of Cl\(^{-}\) deoxygenation increases, which destroys the corrosion layer. Then, the electrolyte slowly flows into the matrix to corrode at the damaged regions, but the corrosion rate is greatly reduced due to the effect of the dense strengthening layer and the passivation film. In WUSGP, an obvious better performance of corrosion resistance was obtained and can be interpreted as follows: the surface matrix undergoes plastic deformation after surface treatment, resulting in an increase in the martensite content with different shapes, such as needle-like and lath-like, which makes the surface structure denser and hinders Cl\(^{-}\) from eroding the matrix. Moreover, the high dislocation density and small grain size can significantly improve corrosion resistance.

4. Conclusions

This paper investigated the corrosion behavior of four GCr15 steel samples in a salt-frog environment. These samples were treated by ultrasonic shot peening (USP), a dry ultrasonic strengthening grinding process (DUSGP), a wet ultrasonic strengthening grinding process (WUSGP), and polishing. These process samples were named Sample polished, USP, DUSGP, and WUSGP, respectively. The capacitance arc, polarization curves, corrosion morphology, and corrosion products were analyzed.

The sample that experienced a surface treatment showed significantly better performance in terms of corrosion resistance, especially the Sample WUSGP, which has the largest capacitive loops. The corrosion potential and corrosion current values are \(-0.3999\) V and \(9.435\) mA/cm\(^2\), respectively. Corrosion morphology revealed that the Sample WUSGP has the smallest corrosion holes and cracks, exhibiting a diameter of approximately 1.4 \(\mu\)m and a width of approximately 2.1 \(\mu\)m, respectively, degrading 78% and 75% compared with the polished sample. Corrosion products analysis showed that the corrosion mainly occurs in the weak regions of the substrate and in the active anode area will produce Fe(OH)\(_2\) film through a redox reaction. The continuous erosion of Cl\(^{-}\) destroyed the corrosion layer. Then, the electrolyte slowly flows into the matrix to corrode at the damaged regions, but the existence of a dense strengthening layer and passivation film greatly reduces the corrosion rate.

The best corrosion resistance obtained in sample WUGP can be interpreted as the high dislocation density, small grain size, needle-like, and lath-like shape of martensite formed in the treatment, which hinders Cl\(^{-}\) from eroding the matrix. This research demonstrated that traditional ultrasonic shot peening fused the strengthened liquid and corundum, and ceramic balls for treating materials can significantly improve the corrosion resistance of steels.

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