Influence of ageing time on microstructure, hardness and corrosion of UNS Zeron 100 super duplex stainless steel

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Abstract. The present study is to investigate the influence of ageing time on the microstructure, hardness and corrosion properties of UNS Zeron 100 super duplex stainless steel. The material was solution treated at 1050°C and water quenched, further the ageing has been carried out at 850°C for 30, 60 and 90 minutes. Hardness test and corrosion test were carried out for both solution treated as well as aged specimens for different ageing period. Results of hardness test indicate an increase in hardness as the ageing period increases. Corrosion test results showed good resistance to corrosion as the ageing time increases. Changes in mechanical properties were observed due to sigma phase formation. Scanning electron microscope (SEM) analysis showed the sigma phase precipitation in duplex stainless steel. EDX analysis shows the consumption of ferrite and formation of chromium which indicates the formation of sigma phase. Optical image analysis showed the increment of sigma phase as the ageing time is increased.

1. Introduction

Duplex stainless steels (DSS) are called “duplex” as they have assemblage of two phases of ferrite and austenite. Zeron 100 (DSS) has found wide industrial application in pharmaceutical, chemical, desalination, pulp, paper industries, power generation and mining industries. The effect of ageing temperature and ageing time effects on a 25Cr-6.7Ni-0.32N-3.0Mo-2.5W (DSS)[1]. The ageing temperature of 600-900°C and ageing time of 1 to 240 minutes which resulted in formation of chi phase and sigma. Ageing significantly influenced on the mechanical properties due to precipitation of inter-metallic phases. The sigma phase effects on Z3CN20M cast (DSS). At temperature of 600-900°C, formation of sigma phase occurs at ferrite phase boundaries[2]. Further, authors have observed that for temperature range less than 700°C and aged for less than 4 hours yield strength and tensile strength was less when compared to aged specimens. Study carried out on SAF2507 SDSS at the temperature range of 200°C-700°C there was no significant change in mechanical properties. And at 800°C formation of sigma phase starts in SDSS[3]. The effects of solution treatments (1020°C 1080°C 1200°C) and different continuous cooling rates (1, 0.5, 0.25 and 0.1°C/second) on 2205 DSS and reported that precipitation of sigma phase was obtained in lower cooling rates and at lower solution treated temperature[4]. It was observed that the sigma phase formation occurs at 850°C significantly high[5]. DSS was heat treated at 700°C and 750°C which resulted the inter-metallic phases which were formed in UNS S31803 DSS. The formation of chi and sigma phase was noticed. It was found that chi phase precipitation was initiated at ferrite/ferrite grain boundaries and chi phase precipitation occurs before sigma phase precipitation[6]. From the above literature it was observed that there is less focus on Zeron 100 SDSS. Hence the present study will give the insight to the influence of ageing on microstructure and mechanical properties of Zeron 100 SDSS.

2. Experimentation

The commercially available material Zeron 100 duplex stainless steel was selected to carry out the experiments to study effect of ageing on Microstructure, Mechanical and Corrosion properties. The prepared specimens for these tests were polished with metallographic techniques[7]. The material was initially solution treated at 1050°C for 120 minutes and quenched in water to obtain the balanced microstructure of austenite and ferrite. The ageing was carried out at 850°C for 30, 60 and 90 minutes and all the specimens were quenched in water. Scanning electron microscope (SEM) was used to
study the microstructure and the corresponding elemental analysis. The hardness test was conducted using Rockwell hardness test rig. Optical image analyzer as shown in Figure 1 was used to study the volume fraction of the phases observed in the material after the ageing. To determine the corrosion resistance of the specimen Salt spray or fog test is conducted in corrosion analyzer as shown in Figure 2.

3. Result and Discussion
The following sections describes results of solution treatment, heat treatment

3.1 Solution Treatment
The solution treatment was carried at 1050°C for 120 minutes. The Figure 3 and Figure 4 are the SEM and optical micrographs of solution treated specimens. It was observed that all the precipitates which exist in the Zeron 100 DSS are dissolved and only equal phase microstructure of ferrite and austenite was obtained. The chemical composition after the solution treatment is presented in the Table 1. The phase volume fraction analysis resulted in 57% of ferrite and 43% austenite for solution treatment.

| Table 1 Chemical Composition of UNS Zeron 100 SDSS |
|----------|----------|----------|----------|----------|----------|----------|----------|
| C        | Mn       | Si        | S        | P        | Cr       | Ni       | Mo       | Cu       | W        | Fe       |
| 0.30     | 0.476    | 0.494     | 0.009    | 0.027    | 25.378   | 7.153    | 3.437    | 0.598    | 0.620    | 61.778   |

3.2 Heat Treatment
The heat treatment was done at 850°C for 30, 60 and 90 minutes and quenched in water. The precipitation of secondary inter-metallic phases was observed. When AISI ZERON 100 SDSS was heated to the formation of sigma phase precipitates could be observed in the microstructure of SDSS.
It precipitates clearly at the austenite and ferrite boundary region and as the aging time increases the ferrite phase of DSS is consumed and sigma phase precipitation increases as observed in the Figure 5 a, b and c for 30, 60 and 90 minutes of ageing time respectively. Figure 6 a, b and c are the phase volume fraction of Zeron 100 duplex stainless steel at ageing time for 30, 60 and 90 minutes respectively. The heat treatment process introduces microstructural changes promoting inter-metallic changes. As the ageing time of heat treatment increases the phase volume of sigma phase increases as observed in the Figure 6 a, b and c. The phase volume fraction of all the three phases for different ageing time is given in the table 2.

| Heat Treatment Temperature 850°C | Phase Volume Fraction (%) |
|----------------------------------|-----------------------------|
| Ageing Time (min)                | Ferrite | Austenite | Sigma |
| 0                                | 56.62   | 43.38     | 0     |
| 30                               | 53.84   | 40.85     | 5.31  |
| 60                               | 46.65   | 45.48     | 7.87  |
| 90                               | 43.07   | 42.40     | 14.53 |

3.3 EDX Analysis results

indicated that there is increase of chromium as the ageing time is increased. Although the increment observed was not much but there was a decrement in ferrite. This is because the sigma phase nucleates at the ferrite phase by consuming chromium and molybdenum. The table 3 shows the chemical composition for different ageing times in terms of weight and atomic mass percentage. It clearly indicates the increase in chromium as the ageing time increases. Result obtained after Optical Image Analysis concludes that as the aging time increases the diffusion rate goes on increasing and hence we can see more ferrite promoting elements gets concentrated and hence sigma phase goes on increasing.

3.4 Hardness

Hardness value has a direct relationship with the sigma phase formation. Specimens were tested for Rockwell hardness value at 850°C for different aging time. Due to the precipitation of inter metallic phase called sigma phase, the Rockwell hardness value is found to be increasing as the aging time increases.
Table 3 Mass percentage of different elements at different ageing time

| Ageing Time(min) | Solution treated specimens | 30 | 60 | 90 |
|------------------|-----------------------------|----|----|----|
| Element          | Weight% | Atomic% | Weight% | Atomic% | Weight% | Atomic% | Weight% | Atomic% |
| Molybdenum       | 3.65    | 1.99    | 3.80    | 2.01    | 3.79    | 1.99    | 3.92    | 2.09    |
| Chromium         | 28.18   | 28.36   | 28.35   | 27.70   | 29.62   | 27.81   | 29.12   | 28.59   |
| Fe               | 60.06   | 56.28   | 58.94   | 53.63   | 58.05   | 52.53   | 58.63   | 53.59   |

Fig.7 Rockwell hardness value for different aging time at 850°C

There is no much difference in the Rockwell hardness number between the aging times, but it is found to be increasing. Figure 7 shows us the Rockwell hardness value for different aging time at 850°C.

3.5 Corrosion

The examinations of corrosion resistance have revealed that the ageing process after super saturation do not cause significant changes in the anti-corrosive properties. The σ phase precipitates occurring along the boundaries of primary solidification grains, according to the reaction δ ferrite→phase+γ’ secondary austenite. The prolonged annealing time results in the ferrite decomposition hence improving the corrosion resistance. The test carried out here showed no formation of white and red rust after 90 min of test as observed in Figure 8 and 9.

Fig.8 Before corrosion treatment
Fig.9 After corrosion treatment
4. Conclusion

- Solution treated Zeron 100 only showed austenite and ferrite phases, however after heat treatment bright sigma phases rich in chromium were formed at the interface of ferrite and austenite phases.
- SEM analysis showed the formation of sigma phase at the interface of ferrite and austenite phases. Optical image analysis revealed the increase in the formation of sigma phase as the ageing time of heat treatment was increased.
- EDX analysis showed the formation of chromium-molybdenum rich phase.
- Due to the formation of sigma phase the hardness value was found to be increasing in the heat treated specimens as compared to the solution treated specimens, this also indicates the reduction in toughness and the brittle nature of Zeron 100.
- The chromium rich phase improved the localized corrosion resistance in heat treated Zeron 100.

5. References

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