Study on Salt Spray Delayed Cracking Performance of Ultra-high Strength Martensitic Steel

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Abstract: The delayed cracking performance of three kinds of automotive ultra-high strength martensite steels, MS980, MS1180 and MS1300, was studied by the salt spray delayed cracking test of deep drawing and punching cup, and the relationship between delayed cracking performance and microstructure was analyzed. The results show that the best of anti-delayed fracture resistance is MS980 steel. The worst of anti-delayed fracture resistance is MS1300 steel. In addition, the results also show that there is a critical forming ratio of three MS steels (MS980 steel is 1.7, MS1180 steel is between 1.5 and 1.6, and MS1300 steel is between 1.3 and 1.4). Below the critical forming ratio, there is no delay cracking in deep drawing specimen. And the critical forming ratio can be used as a basis for evaluating the delay fracture of ultra-high-strength steel under some condition.

1. introduction

With the development of equipment manufacturing industry, especially military industry, the strength of steel is getting higher and higher. However, when the tensile strength of steel exceeds 1200 MPa, delayed cracking is easy to occur\textsuperscript{[1]}. Delayed cracking is a brittle cracking with no obvious macroscopic plastic deformation and no warning before cracking, so it is extremely harmful. Therefore, it is generally necessary to evaluate the delayed cracking performance of ultra-high strength steel before it is used. At present, the accelerated delayed cracking evaluation methods adopted by the laboratory are roughly:
constant load test, slow strain rate tensile test (SSRT), constant strain (bending beam) test and fracture mechanics test, but these delayed cracking evaluation methods have shortcomings, and there is no unified and accepted reasonable evaluation method\textsuperscript{[2-6]}. Therefore, it is of great significance for the development of ultra-high strength steel to explore a set of rapid and accurate evaluation methods for delayed cracking.

In this paper, the delayed cracking performance of three kinds of automotive ultra-high strength martensite (MS) steels, MS980, MS1180 and MS1300, was studied by the salt spray delayed cracking test of deep drawing and punching cup, and the relationship between delayed cracking performance and microstructure was analyzed. At the same time, the feasibility of the salt spray delayed cracking test of deep drawing cup as the evaluation of ultra-high strength steel delayed cracking is discussed, which provides a certain reference for the evaluation of ultra-high strength steel delayed cracking.

2. Test materials and methods
The grades of three kinds of ultra-high strength martensite (MS) steels are: MS980, MS1180 and MS1300, and their specific chemical compositions are shown in Table 1.

| Steels  | C  | Mn | Si  | Al  | Nb+V+Ti | Fe   |
|---------|----|----|-----|-----|---------|------|
| MS980   | 0.11 | 1.72 | 0.44 | 0.040 | <0.055 | Bal. |
| MS1180  | 0.14 | 1.75 | 0.49 | 0.049 | <0.050 | Bal. |
| MS1300  | 0.15 | 1.70 | 0.40 | 0.044 | <0.050 | Bal. |

Samples were taken from the side of the test steel in the rolling direction at the front of drawing and punching cup forming, and metallographic samples were prepared, which were corroded by 4% nitric alcohol solution. The microstructure of steel was observed by Zeiss EVO 15 scanning electron microscope (SEM).

The forming of deep drawing and punching cup samples is carried out according to the national standard GB/T 15825.3-2008"formability and test methods of sheet metal part 3: deep drawing and deep drawing load test"\textsuperscript{[7]}, with punch diameter $d_p=50$ mm, and five parallel samples of each steel under the same forming ratio (ratio of sample diameter to $d_p$ before forming). The salt spray delayed cracking test lasts for 7 days, first the salt spray condition is 24 hours, then the dry-wet cycle (8 hours under wet-hot condition+16 hours under dry condition) is 96 hours, and finally the dry condition is 48 hours. The salt spray delayed cracking test needs 10 cycles for 70 days (1680 h), and the salt spray solution is 5%NaCl aqueous solution.

3. Test results and analysis
3.1. Microstructure
The microstructure of ultra-high strength martensite (MS) steel is shown in fig.1, and the microstructure of three MS steels is martensite (M)+ ferrite (F). Martensite (M) is a strip-shaped region, which is convex because of its high carbon content and is not easy to be corroded by 4% nitric alcohol. The ferrite (F) is a polygonal block area, which is easily corroded by 4% nitric alcohol because of its relatively low carbon content, so it is in a concave state. Martensite, as a strengthening phase, is dispersed on polygonal ferrite matrix with good plasticity in island shape. In addition, it can be seen from the figure that with the increase of the strength grade of the test steel, the microstructure of the test steel is refined and the martensite volume fraction increases obviously, among which the grain size of the MS1300 steel is the smallest and the martensite volume fraction is the highest.
3.2. Test results and analysis

Fig. 2 shows the test results of three kinds of MS steel punching cup samples after 70 days (1680 h) salt spray test. The black square point in the figure is the cracking time of each sample, and the red line is the cracking time of linear fitting. It can be seen from Fig. 2a that after the test is finished, there is no cracking phenomenon when the forming ratio of MS980 steel punching cup sample is 1.7, one cracking sample appears when the forming ratio reaches 1.8, and all samples appear cracking when the forming ratio is 1.9; There is no cracking phenomenon when the forming ratio of MS1180 steel punching cup sample is 1.5, but there is cracking sample when the forming ratio reaches 1.6 (one of which has a cracking time of 1656h, which is close to the end time of the test), and all the samples have cracking when the forming ratio is 1.8, and the cracking time is shorter than that of MS980 steel, as shown in Fig. 2b; The MS1300 steel punching cup sample has no cracking phenomenon when the forming ratio is 1.3, while the forming ratio reaches 1.4, which is a partially cracked sample (one of which has a cracking time of 1586h, which is close to the end of the test), and the forming ratio is 1.7, which is shown in Fig. 2c. It shows that among the three ultra-high strength martensitic steels, MS980 steel has the best delayed cracking resistance, followed by MS1180 steel, and MS1300 steel has the worst delayed cracking resistance. Generally speaking, the higher the strength grade of the test steel, the more likely it is to have delayed cracking\cite{2,8}. In addition, from Figure 2, it can be found that the larger the forming ratio of the same test steel punching cup sample, the more cracks appear, and the shorter the cracking time. This shows that the same test steel is more likely to crack under high forming ratio, which is because the greater the forming ratio of the punching cup sample, the greater the internal stress of the sample, and the greater the possibility of delayed cracking under the same conditions.
Fig. 2 shows the result of salt spray test for steels (a) MS980; (b) MS1180; (c) MS1300.

Fig. 3 shows the cup-punching samples of three ultra-high strength dual-phase steels after salt spray test. As shown in Fig. 3a, after 70 days of salt spray test, the sample surface of MS980 steel is seriously corroded, and there are many corrosion products, which are mainly rust from the appearance, and the sample (forming ratio is 1.8) has obvious cracking phenomenon at the cup mouth. After salt spray test for 70 days (or cracking time), the surface of MS1180 steel is seriously corroded, and there are many corrosion products such as rust. The samples with high forming ratio (1.6, 1.7 and 1.8) have obvious cracking phenomenon at the cup mouth, as shown in Fig. 3b. After salt spray test for 70 days (or cracking time), the surface of MS1300 steel is seriously corroded, and there are many corrosion products such as rust, etc., and obvious cracking phenomenon appears at the cup mouth when the forming ratio is above 1.4 (Fig. 3c). It can also be seen from fig. 3 that the crack is wide at the mouth of the cup, and extends to the bottom of the cup, and the crack is flat. Cracks appear suddenly, and the incubation period of cracks may be long but the propagation speed is fast. Combined with these characteristics, it can be determined that it is brittle cracking, which basically belongs to delayed cracking, rather than corrosion cracking caused by corrosion.
It can be found from fig.2 that with the decrease of the forming ratio of the three MS steels, the cracking time of the samples becomes longer, and when a certain critical forming ratio (MS980 steel is 1.7, MS1180 steel is between 1.5 and 1.6, and MS1300 steel is between 1.3 and 1.4), the cup-punching samples will not crack, which is the same test rule. This test rule is somewhat similar to fatigue test, The ordinate of fatigue test is the stress amplitude of cyclic stress (corresponding to the salt spray test is the molding ratio of cup punching test), and the abscissa is the cycle number (corresponding to the salt spray test time). In a certain specified cycle number (generally 107), fatigue fracture will not occur when the stress amplitude of cyclic stress is lower than the critical stress value, which is the conditional fatigue limit. Three kinds of MS steels also have similar conditional delayed cracking limits, that is, within a specified time of salt spray test (70 days, 1680h in this paper), when the critical forming ratio (MS980 steel is 1.7, MS1180 steel is between 1.5 and 1.6, and MS1300 steel is between 1.3 and 1.4) is below, delayed cracking will not occur. That is to say, the critical forming ratio of ultra-high strength steel in salt spray test can be used as the evaluation basis of whether it will have delayed cracking under certain conditions. However, in this paper, under certain conditions, it is assumed that after 70 days (10 cycles) of salt spray test, the steel punching cup sample will not crack, and it is considered that the test steel will not have delayed cracking during its entire 20-year service life (the average service life of ordinary automobiles will not exceed 20 years). At present, our existing research results show that it is assumed that there is no cracking phenomenon in the steel punching cup sample in one cycle salt spray test, and that there is no delayed cracking phenomenon in the test steel within two years or even longer. According to the results of delayed cracking test of punching cup in atmospheric environment at present, after 4 years, we found that only one sample of MS1180 steel with forming ratio of 1.7 cracked in 18672 h (about 2 years and 48 days), and the critical forming ratio of MS1180 steel was between 1.5 and 1.6, which proved that the critical forming ratio of delayed cracking assumed by us was effective. Of course,
more time and more in-depth study are needed to determine whether this hypothesis is valid in a longer period of time.

In addition, from fig. 1, it can be found that the volume fraction of martensite in the microstructure of MS980, MS1180 and MS1300 steel is gradually increased, and the strength grade of the test steel is also gradually improved, and the sensitivity of delayed cracking of the test steel is also higher. This shows that the existence of martensite in the test steel is not conducive to improving the delayed cracking performance of the test steel, but is beneficial to improving its strength, which requires us to select materials to balance the relationship between strength and delayed cracking. Martensite volume fraction may have a critical value for delayed cracking performance of test steel, When the martensite volume fraction in test steel is lower than the critical value, it will not cause delayed cracking phenomenon. Of course, the more detailed relationship between them needs more in-depth study.

4. Conclusion

(1) The microstructure of three ultra-high strength martensitic steel is martensite+ferrite, and the volume fraction of martensite increases obviously with the increase of strength grade of test steel.

(2) The results of salt spray delayed cracking test of deep drawing punching cup show that MS980 steel has the best delayed cracking resistance, followed by MS1180 steel, and MS1300 steel has the worst delayed cracking resistance. The larger the forming ratio of the same test steel punching cup sample is, the more cracks appear and the shorter the cracking time is.

(3) The test results show that there is a critical forming ratio (1.7 for MS980 steel, 1.5-1.6 for MS1180 steel and 1.3-1.4 for MS1300 steel) in the three kinds of MS steels, When the critical forming ratio is lower than this, there will be no delayed cracking phenomenon in the punching cup samples, and this critical forming ratio can be used as an evaluation basis for whether the ultra-high strength steel will have delayed cracking under certain conditions.

Author brief introduction

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