THE EFFECT OF COLD WORK ON THE SENSITISATION OF AUSTENITIC STAINLESS STEELS

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1 INTRODUCTION

The sensitisation behaviour of austenitic stainless steel is greatly influenced by several metallurgical factors, such as the chemical composition, the degree of prior deformation, the grain size, and the ageing temperature and time. The precipitation behaviour of AISI 316 and 304 austenitic stainless steels has been investigated after ageing at various temperatures from 500 °C to 900 °C for 0.1 h to 1000 h. The TTS diagrams of the experimental steels after an oxalic-acid etch test ASTM A262 practice A were constructed. It was demonstrated that the C curves of the TTS diagrams were displaced towards shorter times by the increment of 20 % cold work (CW), since the sites inside the grain matrix have a high energy and the carbides can nucleate there easily. Cold work increases the number of dislocations/dislocation pipes along which the diffusion rate of chromium is very high. The sensitisation of the experimental steels accelerated the precipitation of M₆C. Besides M₆C, the σ-phase and M₆C were detected at the grain boundaries and in the austenitic matrix in the case of the cold-worked samples.

Therefore, it has been suggested that the nature of grain boundaries could also influence the DOS and IGC.

In this article we report on some preliminary comparisons of the combined effects of chemical composition, deformation, temperature and aging time on sensitisation in AISI 304 and 316 stainless steels.

2 MATERIALS AND EXPERIMENTAL PROCEDURES

The chemical composition of the experimental steels is given in Table 1. The steels were mostly investigated in the as-received condition with some in the solution-annealed condition. The solution annealing was conducted on the as-received materials at 1050 °C for 60 min followed by water quenching.

The steels were 20–40 % cold rolled by controlling the thickness of the plates. The cold-worked samples were heat treated at various temperatures in the range 500–900 °C for times of 0.1 to 1000 h. The samples were then water quenched after the heat treatment.

The oxalic-acid etch test (ASTM A262 practice A) was used to determine the steels’ sensitivity to intergranular corrosion. The specimens were electrolytically etched in 10 % oxalic acid for 90 s at a current density of 131
1 A/cm². The etched microstructure was then examined at 250x, and was characterised as a step, dual or ditch microstructure.

For the individual secondary-phase identification transmission electron microscopy (TEM) of the carbon extraction replicas was applied. TEM observations were performed using a JEOL 200 CX operating at 200 kV. The carbon extraction replicas were obtained from mechanically polished and etched surfaces. The replicas were stripped from the specimens in the solution of CH₃COOH : HClO₄ = 4:1 at 20°C and 20 V.

### 3 RESULTS

The results of the light microscopy examination are summarised in Figure 1. The microstructure of AISI 304 after solution annealing consists of polyhedral austenitic grains with twinning typical of an fcc microstructure. The average austenitic grain size in this state is about 45 µm (Figure 1a). A small amount of δ-ferrite was also observed. No precipitates were detected at the grain boundaries (GBs) of the solution-annealed steels. Figure 1b shows the microstructure of the AISI 304 after 40 % of CW. The microstructures of the aged states are shown in Figure 1c and Figure 1d. Figure 1c shows the evolution of secondary phases precipitated at the GB in the isothermally aged specimen (650 °C/0.5 h) without cold work. The microstructure of the isothermally aged specimen (650 °C/0.5 h) and 40 % CW is shown in Figure 1d. The precipitation of secondary phases was observed at the GB and intragranularly and within the matrix.

To compare the results of two austenitic stainless steels, time-temperature-sensitisation (TTS) diagrams for these steels for different degrees of CW ranging from 0 % to 40 % are presented in Figure 2. From the TTS diagrams it can be seen that the nose of the C curve corresponding to the maximum rate of sensitisation
occurs at 800 °C for the AISI 316 in the 0 % CW condition. As the degree of CW increases, the nose temperature remains almost that same, but $t_{\text{min}}$ decreases with the increase in % CW up to 20 % and remains constant thereafter. The TTS diagram of AISI 304 is shifted towards shorter times than the 0 % CW material. The tendency of the shift of the AISI 304 C curve is similar to the case of AISI 316.

To identify the type of secondary phases precipitated at the grain boundaries (GBs) during the isothermal treatment, TEM analysis was carried out. First, $M_23C_6$ was detected at the grain boundaries after aging. In addition to $M_23C_6$, the $\sigma$-phase and $M_6C$ were detected at the grain boundaries (Figure 3 and Figure 4). Similar precipitation trends were detected using TEM analysis at
the grain boundaries of the AISI 304 steel. The identified secondary phases in the experimental steels and the phase ratio are shown in Figures 5 and 6.

4 CONCLUSIONS

The precipitation behaviour of AISI 316 and 304 austenitic stainless steels was investigated during aging at various temperatures in range from 500 °C to 900 °C for times from 0.1 to 1000 h. The following conclusions can be drawn:

TTS diagrams of the experimental steels after the oxalic-acid etch test ASTM A262 practice A show that the C curves of the TTS diagrams are displaced towards shorter times with increasing amounts of CW.

After ≈20 % cold working, even the sites inside the grain matrix have high energy and carbides can nucleate there also. Cold work increases the number of dislocations/dislocation pipes along which the diffusion rate of chromium is faster.

Sensitisation of the experimental steels accelerated the precipitation of M_{23}C_6. In addition to this carbide, σ-phase and M_6C were detected at the grain boundaries and in the austenitic matrix in the cold-worked samples.

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5 REFERENCES

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