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# **EFFECT OF ETCHING METHODS IN METALLOGRAPHIC STUDIES OF DUPLEX STAINLESS STEEL 2205**

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Three different etching methods are used to uncover the ferrite-austenite structure and precipitates of secondary phases in stainless steel 22.5% Cr – 5.4% Ni – 3% Mo – 1.3% Mn. The structure is studied under a light microscope. The chemical etching is conducted in a glycerol solution of  $HNO<sub>3</sub>$ , HCl and HF; the electrochemical etching is conducted in solutions of KOH and NaOH.

*Key words:* duplex stainless steel; secondary phases, etching, light microscopy.

## **INTRODUCTION**

Duplex stainless steels (DSS) are often treated for forming a balanced structure containing about 50% austenite and 50% ferrite. This provides the desired combination of hardness, toughness and corrosion resistance  $[1 - 4]$ . These steels possess characteristics of both ferrite (high strength, corrosion resistance, enhanced endurance) and austenite (high general corrosion resistance). The strength of DSS is about 1.5 times higher than that of austenitic stainless steels, and the high corrosion resistance is combined in them with high resistance to pitting corrosion and stress corrosion cracking. This widens the application of these steels in the oil, chemical, petrochemical, nuclear and marine industries. The composition, heat treatment and deformation treatment determine the phase transformations occurring in the steels, their mechanical and corrosion properties and the mode of processing  $[5 - 7]$ . However, when a DSS is subjected to a long-term impact of high temperatures  $(650 - 950^{\circ}$ C), its structure may acquire several undesirable intermetallic phases. As a rule, the intermetallic phases are enriched with chromium and molybdenum as compared to the nominal composition and grow from the ferritic phase, which is also enriched with these elements. The intermetallic phases influence negatively the mechanical properties and the corrosion resistance of DSS  $[7 - 13]$ . It is important to determine optimum heat treatment conditions for controlling the volume fraction of these phases [7], which are best detectable by metallography.

The aim of the present work was to study the etching behavior of duplex stainless steels for determining their ferrite-austenite structure and precipitation of secondary phases.

#### **METHODS OF STUDY**

We studied specimens  $15 \times 10 \times 10$  mm in size fabricated from duplex stainless steel SAF 2205 (1.4462) with the following chemical composition (in wt.%): 0.02 C, 22.56 Cr, 5.42 Ni, 2.95 Mo, 1.29 Mn, 0.57 Si, 0.031 P, 0.014 S, 0.170 N, the remainder Fe.

The furnace in the heat treatment was heated at a constant rate of  $8 \text{ K/min}$  to  $1050^{\circ}\text{C}$ ; the steel specimens were held at this temperature for 1, 8 and 10 h. For each condition, the specimen was either quenched in water of cooled with the furnace. The metallographic study of the secondary phases was performed in sections transverse to the rolling direction**.** The laps were prepared with the help of air-cooled silicon carbide abrasive papers with grit sizes 180, 240, 320, 400, 600, 800, 1000, 1200 and 2000. Then the laps were polished with a diamond paste with particle size  $1 \mu m$ . The etching was performed by three methods, i.e., chemical etching in a glycerol solution of  $HNO<sub>3</sub>$ , HCl, and HF and electrochemical etching in a 50% solution of KOH (5 V for  $4 - 8$  sec) and a 20% solution of NaOH (8 V,  $4 - 8$  sec). The structure of the specimens was examined under an optical microscope (OM).

#### **RESULTS AND DISCUSSION**

Figure 1 presents the microstructure of specimens of alloy SAF 2205 after 1-h annealing at 1050°C and cooling with the furnace, which has been uncovered by three variants of etching. The austenite has the form of islands in the ferrite matrix. Particles of secondary phases are absent in Fig. 1*a* and *b*. In Fig. 1*c* we can see dark inclusions of secondary

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phases on ferrite/austenite boundaries detectable after electrochemical etching in a 50% solution of KOH.

Figure 2 presents the microstructure of specimens annealed at 1050°C for 8 and 10 h and cooled with the furnace. Like in Fig. 1, the austenite is represented by islands in the ferrite matrix. Secondary phases are not detectable after chemical etching and after electrochemical etching in a 20% solution of NaOH. Dark etched secondary phases are observable on austenite/ferrite boundaries after electrochemical etching in a 50% solution of KOH (Fig. 2*c* and *f* ).

Chemical etching in a glycerol solution of  $HNO<sub>3</sub>$ , HCl and HF and electrochemical etching in a 20% solution of NaOH uncovers a ferrite-austenite microstructure. However, these methods of etching do not show secondary phases (Figs. 1 and 2) even when their volume fraction is large and the duration of the annealing is prolonged. Secondary phases in duplex steel SAF 2205 are detectable only by electrochemical etching in a 50% solution of KOH.

The austenite-ferrite transformation in duplex steel SAF 2205 occurs above 1000°C and develops with prolongation of the hold. When the specimens are cooled, the ferrite transforms into austenite and intermetallic phases by a eutectoid reaction. Therefore, the content of intermetallic phases is the higher the larger the volume fraction of ferrite.

Figure 3 presents the microstructures of specimens after annealing for different times and cooling with the furnace. The structure is un-



**Fig. 1.** Microstructure of steel SAF 2205 after 1-h annealing at 1050°C and cooling with the furnace (the arrows point at intermetallics): *a*) chemical etching; *b* ) electrochemical etching in 20% solution of NaOH; *c*) electrochemical etching in 50% solution of KOH.





**Fig. 2.** Microstructure of steel SAF 2205 after annealing at 1050°C for 8 h  $(a-c)$  and 10 h  $(d-f)$  and cooling with the furnace (the arrows point at intermetallics): *a*, *d* ) chemical etching; *b*, *e*) electrochemical etching in 20% solution of NaOH; *c*, *f* ) electrochemical etching in 50% solution of KOH.



**Fig. 3.** Microstructure of steel SAF 2205 after annealing at 1050°C for  $1 \text{ h } (a)$ ,  $8 \text{ h } (b)$  and  $10 \text{ h } (c)$  and cooling with the furnace (the arrows point at intermetallics). Electrochemical etching in 50% solution of KOH.

covered by electrochemical etching in a 50% solution of KOH. Whatever the duration of the annealing, the secondary phases are arranged on grain boundaries of ferrite and on ferrite/austenite boundaries. The secondary phases in DSS consist of iron, chromium and molybdenum. Chromium and molybdenum are ferrite-stabilizing elements, and their content in the ferrite is higher than in the austenite. In this connection, the secondary phases in the DSS grow to the depth of the ferrite phase, as it has been shown in [14]. The size of the crystallites of the secondary phases increases with the time of the annealing. The fraction of the ferrite phase also grows with increase in the annealing time due to the  $\alpha \rightarrow \gamma$  transformation. Under cooling, the growth of the secondary phases is intensified with increase in the time of annealing, because the contents of chromium and molybdenum in the ferrite is elevated.

#### **CONCLUSIONS**

1. Electrochemical etching in a 50% solution of KOH uncovers secondary intermetallic phases in duplex stainless steel SAF 2205. Chemical etching in a glycerol solution of  $HNO<sub>3</sub>$ , HCl and HF and electrochemical etching in a 20% solution of NaOH uncovers a ferrite-austenite microstructure.

2. Etching in the solutions tested does not reveal secondary phases except for the solution of KOH.

3. Prolongation of annealing at 1050°C increases the volume fractions of ferrite and secondary phases and the size of the crystallites of the secondary phases.

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