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# Effect of cold working and isothermal aging on the precipitation of sigma phase in 2205 duplex stainless steel

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## ABSTRACT

A comprehensive understanding of the formation of the sigma phase resulting from the eutectoid reaction ( $\delta \rightarrow \sigma + \gamma_2$ ) is required. The kinetics necessary for the eutectoid reaction are closely related to the amount of plastic deformation in DSS. This work investigates the microstructural evolution of the  $\sigma$  phase in a 22Cr–5Ni SAF 2205 DSS after subsequent plastic deformation and isothermal aging. The precipitation of the  $\sigma$  phase resulted from the higher driving force for precipitating intermetallic compounds and the higher diffusion rate of their elements by cold-rolling, as well as the sufficient supply of molybdenum. The maximum amount of  $\sigma$  phase precipitation remarkably increased with an increasing cold deformation, as compared with the non-cold-rolled materials.

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## 1. Introduction

Duplex stainless steel (DSS) has been extensively used in many applications because of its excellent mechanical properties and corrosion resistance. A number of studies have been conducted on the kinetics and microstructural evolution of the secondary phases in DSS, since the aging of DSS leads to a series of phase transformations that take place in the ferrite or at the grain boundaries or  $\delta/\gamma$  interfaces [1–4]. In a previous study [5], the nucleation and growth of  $M_{23}C_6$  carbides caused the migration of the  $\delta/\gamma$  interface boundary into the ferrite phase region. Badji et al. [6] showed that the  $\sigma$ -phase and  $M_{23}C_6$  carbides were precipitated at the  $\delta/\gamma$  interfaces and within the  $\delta$ -ferrite grains.

The precipitation of phases such as the  $\sigma$  phase, secondary austenite,  $\chi$  phase, and carbides after plastic deformation has recently received considerable attention, because the presence of such precipitates significantly influences the mechanical properties and corrosion resistance of DSS. Several researchers [7–9] reported that plastic deformation in DSS had a significant

influence on its phase transformations and mechanical, corrosion and formability properties. In another study, the effect of cold-deformation on spinodal ferrite separation was reported [10]. The lattice defects introduced by deformation can enhance the diffusion in the  $\delta$  and  $\gamma$  phases and act as the nucleation sites for  $\sigma$  phase precipitation [11]. Furthermore, the decomposition of the ferrite phase was significantly accelerated by the strain introduced during cold deformation in a nitrogen-alloyed DSS [12].

Because of the important effects of sigma precipitation on the mechanical properties of DSS, a comprehensive understanding of the formation of the sigma phase resulting from the eutectoid reaction ( $\delta \rightarrow \sigma + \gamma_2$ ) is required. The kinetics necessary for the eutectoid reaction are closely related to the amount of plastic deformation in DSS. As such, this work investigates the microstructural evolution of the  $\sigma$  phase resulting from the eutectoid decomposition of the delta ferrite in a 22Cr–5Ni SAF 2205 DSS after subsequent plastic deformation and isothermal aging.

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**Table 1 – Chemical compositions of duplex stainless steels used in the present study (wt.%).**

Steel	C	Si	Mn	Cr	Mo	Ni	V	Co	Cu	N
SAF 2205	0.028	0.24	1.83	21.25	2.600	5.250	0.050	0.040	0.100	0.16

## 2. Materials and Methods

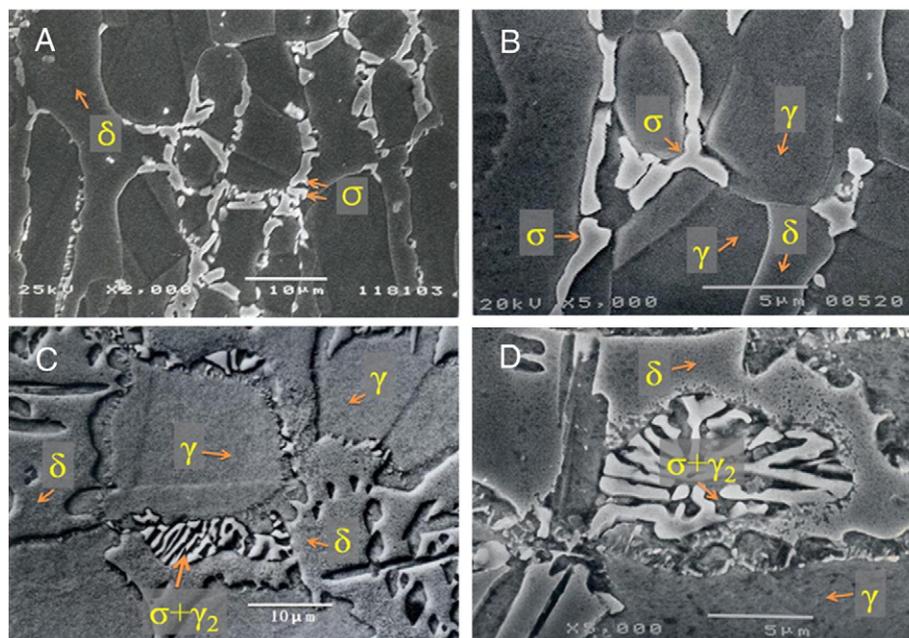
The DSS used in this study was type 2205 with the composition 22Cr–5Ni–3Mo–0.16N (wt.%) and supplied by Böhler Steel Co. (Germany). This material was fabricated into hot-rolled plates with a thickness of 5 mm. The final composition of the SAF 2205 is listed in Table 1. The specimens were solution-treated at temperatures of 1050 °C and 1250 °C, which were chosen based on the isotherms of Fe–Cr–Ni systems [13]. The austenite volumes measured by an image analyzer were 44% and 30% at 1050 °C and 1250 °C, respectively. After solution treatment, the specimens were cold rolled with reductions of 25, 50, and 75%. Then, isothermal aging was carried out at 800 °C for 30, 60 and 120 min.

Optical microscopy and scanning electron microscopy (SEM) were performed after etching in (30 ml HCl+30 ml HNO<sub>3</sub>+45 ml Glycerol+a little HF) reagent. The thin foils used for observation by transmission electron microscopy (TEM) were prepared from the long transverse section parallel to the rolling direction of the cold rolled plates and by twin-jet electropolishing in a solution of 10% perchloric acid, 20% glycerol and 70% ethanol at –10 °C and 20 V. The foils were examined using JEM 2000 FX II TEM equipment operated at 200 kV. The crystallographic analyses of the precipitates and the matrix were performed using the selected area diffraction patterns (SADPs), and an energy dispersive X-ray spectrometer (EDX) was used for the precise identification of each phase.

## 3. Results and Discussion

The SEM micrographs of the specimens aged at 800 °C for 1 h after being solution-treated at 1050 °C and 1250 °C are shown in Fig. 1. This figure shows that there are significant differences in the morphologies of the  $\sigma$  phases at the different solution treatment temperatures. For the specimens solution-treated at 1050 °C, the  $\sigma$  phase is mainly nucleated and grown at the  $\delta/\gamma$  interface. However, in the specimens solution-treated at 1250 °C, the formation of  $\sigma$  phase mainly results in the decomposition of ferrite into a eutectoid structure of  $\sigma$  phase and secondary austenite ( $\gamma_2$ ). The formation of the  $\sigma$  phase by the eutectoid reaction ( $\delta \rightarrow \sigma + \gamma_2$ ) dominantly occurred with an increasing solution treatment temperature. This can be understood on the basis that the driving force for  $\gamma$  formation increases, because the duplex structure is quenched from a higher temperature at which the equilibrium fraction of  $\gamma$  phase is lower. Thus, when the specimen solution-treated at the higher temperature (i.e., 1250 °C) is aged at a lower temperature, the equilibrium fraction of  $\gamma$  becomes higher again. In addition, the driving force for  $\gamma$  formation results in the Widmanstätten precipitation of  $\gamma$  in the ferrite grain. A previous study reported that the  $\gamma$  phases spread into the  $\delta$  phase around the  $\sigma$  phases [5].

Fig. 2 shows the microstructures of SAF 2205 cold-rolled at room temperature after being solution-treated at 1250 °C for 30 min with various deformation ratios of (a) 0%, (b) 25% and (c) 50%. As shown in this figure, the morphology of the



**Fig. 1 – SEM micrographs of SAF 2205 aged at 800 °C for 1 h as a function of solution treatment temperature; (A) and (B):1050 °C, (C) and (D):1250 °C.**

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