# The effect of Cu on R Phase Precipitation and Mechanical Properties in Super Duplex Stainless Steels

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#### Abstract

In this study, we investigated the precipitation behavior of the R-phase precipitated at the initial stage of aging and its effects on the mechanical properties 25%Cr-7%Ni-4%Mo-0.12%Cu,1.63%Cu super duplex stainless steel. The R-phase was mainly precipitated at the interface of ferrite austenite phases and inside of the ferrite phase during the initial stage of aging. It was transformed into the  $\sigma$ -phase with an increase of the aging time. The ferrite phase was decomposed into a new austenite phase and the  $\sigma$ -phase by an aging treatment. The R phase was intermetallic compound showing higher molybdenum and tungsten concentrations than the matrix and also showed higher molybdenum and tungsten concentrations than the  $\sigma$  phase. In the initial stage of aging, precipitation of the R phase did not change the hardness, the strength and the elongation. The hardness and the strength increased upon a longer aging time, but the elongation rapidly decreased. These results show that the R phase did not significantly affect the hardness and the strength, though it did influence the elongation.

**Keywords** – hardness, super duplex stainless steel, tensile properties, R phase,  $\sigma$  phase

# I. INTRODUCTION

Super duplex stainless steels have a value of PREN(pitting resistance equivalent number) over 40, and it is the steel that more improved corrosion resistance and mechanical properties than the original stainless steels. It is widely used in a very poor environment such as in oil refining business, development, and offshore plant business [1-2]. In super duplex stainless steels, 2-4% of Mo is generally added to improve fitting corrosion resistance and strength. However, when Mo is refined or used in high temperature or is gone through aging treatment, there have been many kinds of research performed to improve the corrosion resistance and toughness by suppressing the important extraction of  $\sigma$  phase that significantly reduces mechanical properties and corrosion resistance of super duplex stainless steels [3-5]. Precipitation behavior among

metals such as R,  $\chi$ ,  $\sigma$  phases which are extracted in super duplex stainless steels becomes different according to refinement, using condition, and the condition of aging treatment [6]. Especially, after R phase among these extraction phases is extracted at an early stage of refinement and aging treatment between 550~650 °C , it metamorphosed to  $\sigma$  phase. But, we expect that it will greatly influence on not only the toughness of materials but also on mechanical properties [7]. However, if aging treatment time gets longer after extraction at an early stage of refinement and aging treatment, R phase metamorphosed to  $\sigma$  phase. In addition, the extraction amount is less, so the research on R phase was not actively performed and there is a shortage of research [8]. Therefore, to expand the range of use of super duplex stainless steels and to secure the stability of usage, various researches related to precipitation behaviour of R phase according to the time of aging treatment at 600 °C by adding Cu0.12%, Cu1.63% to super duplex stainless steels and the influences of precipitation behaviour of R phase on impact toughness.

#### **II. MATERIALS AND EXPERIMENTAL APPARATUS**

A sample used in this research is made with small-sized ingot after dissolving at high-frequency vacuum melting furnace. This ingot went through hot rolling between  $1000 \sim 1250 \,^{\circ}{\rm C}$  and is made with a 15mm thick board. The chemical composition of this sample is shown in Table 1 and 2.

С	Ν	S	Mn	Cr	Si	Ni	Mo	Cu			
0.024	0.25	0.004	0.83	24.67	0.4	6.66	3.73	0.12			

 Table 1 Chemical composition (wt.%)

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С	Ν	S	Mn	Cr	Si	Ni	Mo	Cu
0.013	0.23	0.007	1.03	24.88	0.4	6.55	3.65	1.63

 Table 2 Chemical composition (wt.%)

After solution treatment which is doing water cooling after maintaining the sample at  $1050 \,^{\circ}$  for 30 minutes, aging treatment went through heat treatment at  $600 \,^{\circ}$  which is an expected temperature that easily extracts R phase and by differing the time between 30 minutes and 100 hours at maximum. Sample's microstructure which goes through solution treatment is observed with an optical microscope, and after solution treatment, the change of sample's microstructure that went through aging treatment is observed with scanning electron microscope (TESCAN VEGA3) that uses backscatter electron. After producing tensile specimens by following the size of ASTM E-8 that has 50mm of parallel portion, tensile test to measure the tensile property performed the test at a crosshead speed of 2mm/min. On the other hand, the impact test was performed at room temperature with an impact tester.

# **III. RESULTS AND DISCUSION**

Fig. 1 is an observation of microstructure with an optical microscope after solution treatment which is water cooling after maintaining super duplex stainless steels which have the composition of 25% Cr-7%Ni-4%Mo-0.12%Cu for 30 minutes at 1050 °C. We know that ferrite(black) and austenite(white) are formed with a duplex structure that exists together with volume rate. On the other hand, it was investigated that the volume rate of a head is 29.99% of ferrite and 70.01% of austenite. Fig. 2 is an observation of microstructure with an optical microscope after solution treatment which is water cooling after maintaining super duplex stainless steels which have the composition of 25% Cr-7%Ni-4%Mo-1.63%Cu for 30 minutes at 1050 °C. We know that ferrite(black) and austenite(white) are formed with a duplex structure that exists together with volume rate. On the other hand, it was investigated that the volume rate. On the other hand, it was investigated that the volume rate. On the other hand, it was investigated that the volume rate. On the other hand, it was investigated that the volume rate. On the other hand, it was investigated that the volume rate of a head is 15.68% of ferrite and 84.32% of austenite.

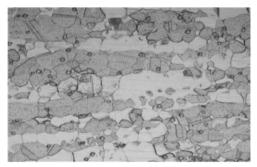


Fig. 1 Optical micrographs of 25%Cr-7%Ni-4%Mo-0.12%Cu

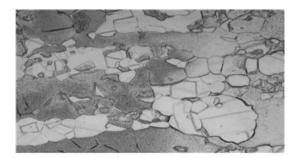
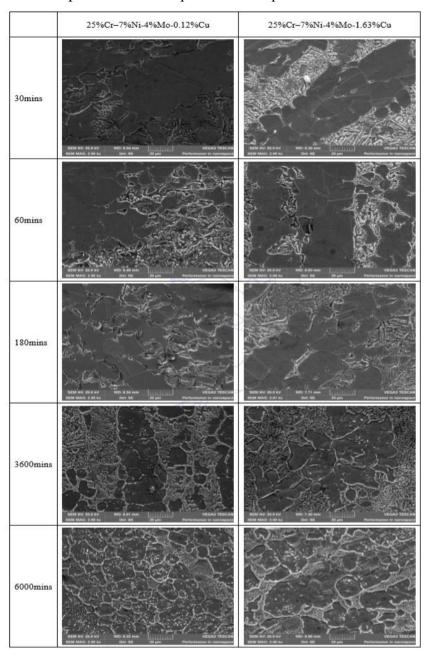


Fig. 2 Optical micrographs of 25%Cr-7%Ni-4%Mo-1.63%

Fig. 3 represents an observation with a scanning microscope that uses backscatter electron to observe the change of microstructure after aging treatment by differing the time at 600°C. After solution treatment, in the sample after aging treatment for 30 minutes, extremely tiny R phases(white) are sequentially extracted following the interface of ferrite and austenite, and they are extracted inside ferrite, too. In the sample after aging treatment for 180 minutes, we knew that the amount of extracted R phases is enormous and the size became bigger. As the time of aging treatment gets longer and becomes 3600 minutes, R phases not only grew significantly, but also the amount is enormous. Parts of ferrite structure are decomposed into the austenite phase and  $\sigma$ 

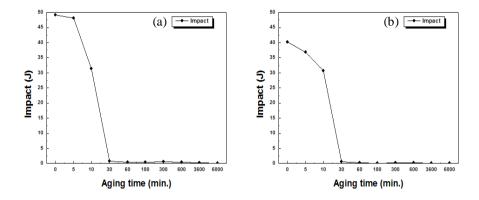
phase, and we know that  $\sigma$  phases are extracted and existed. As the time of aging treatment gets even longer and becomes 6000 minutes, we know that parts of ferrite mostly metamorphosed into the austenite phase and  $\sigma$  phase. Also, the amount of the  $\sigma$  phase became a lot. However, the amount of R phase extracted at an early stage of aging treatment became greatly less, and the reason for the amount of R phase becomes less is considered that R phase is metamorphosed into  $\sigma$  phase.



**Fig. 3** SEM micrographs showing the effect of aging time 25%Cr-7%Ni-4%Mo-0.12%Cu, 1.63%Cu super duplex stainless steel, aged at 600°C for various time after solution treatment at 1050°C 30, 60, 180, 3600, 6000mins

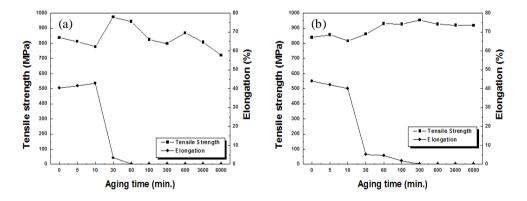
Fig. 4 represents an impact test of a sample that was gone through aging treatment by different the time at 600°C. There was no significant change in impact value at an early stage of aging treatment, but after 30 minutes of aging treatment, impact value rapidly reduces because R and  $\sigma$  phases are resulted to extract.

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**Fig. 4** Effect of aging time the impact in 25%Cr–7%Ni-4%Mo-0.12%Cu(a), 1.63%Cu (b)super duplex stainless steel, aged at 600oC for various time after solution treatment at 1050°C 30mins.

Fig. 5 represents the effect of time of aging treatment on tensile strength and elongation rate. For tensile strength and elongation percentage, there was no significant change at an early stage of aging, but as the time of aging treatment is resulted to extract R and  $\sigma$  phases after 30 minutes, tensile strength gradually increases, and elongation rate rapidly reduces which shows the closer value to 0. After that, there was no big change shown. There is no significant change in impact value, tensile strength, and elongation rate at an early stage of aging, but as the time of aging treatment increases after 30 minutes, tensile strength gradually increases after 30 minutes, tensile strength reduces after 30 minutes at an early stage of aging, but as the time of aging treatment increases after 30 minutes, tensile strength gradually increases while impact value and elongation rate rapidly reduce.



**Fig. 5** Effect of aging time the tensile strength, elongation 25%Cr-7%Ni-4%Mo-0.12%Cu(a), 1.63%Cu (b)super duplex stainless steel, aged at 600°C for various time after solution treatment at 1050°C 30mins.

#### **IV. CONCLUSION**

In this paper, the results can be summarized as follows: (1) R phase is mainly ferrite phase at an early stage of aging, and it is extracted in the interface of the austenite phase and inside ferrite. As it increases along with increasing the time of aging treatment and the time of aging treatments gets longer, it reduces because it metamorphosed to  $\sigma$ phase. (2) As the time of aging treatment gets longer, ferrite phase decomposed into new austenite phase and  $\sigma$  phase. (3) There was no significant change of impact value, tensile strength, and elongation rate at an early stage of aging treatment that extracts R phase, but with increasing the time of aging treatment, while the value of tensile strength gradually increases, impact value and elongation rate rapidly reduced. (4) Tensile strength does not get a big impact on the R phase extracted at an early stage of aging, but impact value and elongation rate were significantly impacted.

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