Effect of R Phase Formation on the Impact Toughness of a 25%Cr–7%Ni–3%Mo Duplex Stainless Steel

Yukio SHIMOIDE, Jie CUI,¹⁾ Chang-Yong KANG²⁾ and Kazuya MIYAHARA³⁾

Daido Institute of Technology, Daido-cho, Minami-ku, Nagoya, Aichi-ken, 467-8531 Japan.

1) Graduate School, Nagoya University, Furoh-cho, Chikusa-ku, Nagoya, Aichi-ken, 464-8603 Japan.

2) Department of Metallurgical Engineering, Pukyong National University, Yong-Dang-Dong, Pusan, Korea.

3) Department of Molecular Design and Engineering, Nagoya University, Furoh-cho, Chikusa-ku, Nagoya, Aichi-ken, 464-8603 Japan.

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The aging behavior (microstructural evolution and mechanical properties change) of a 25%Cr–7%Ni–3%Mo duplex stainless steel at 873 K was investigated. A very short term (3.6 ks) aging treatment caused a significant decrease of impact toughness before the formation of σ phase. Quasi-stable R phase was observed to form locally in the material aged for 3.6 ks. These results indicate that the significant loss of impact toughness due to the R phase formation at the very early stage of aging occurs before the embrittlement due to the σ phase.

KEY WORDS: duplex stainless steel; σ phase; R phase; embrittlement; impact toughness; aging treatment; precipitation.

1. Introduction

Duplex (austenitic-ferritic) stainless steels have superior properties of corrosion resistance, tensile strength, toughness, *etc.* and they are widely used for the structural and component materials of chemical plants. It is well known, however, that σ phase formation due to the decomposition of α phase after the aging treatment at high temperature induces the deterioration of impact toughness.^{1–6)} On the other hand, Nilsson *et al.*⁷⁾ recognized the formation of R phase after the aging treatment at 873 K for 86.4 ks and they estimated the possibility of embrittlement due to the R phase formation. In this research, the present authors have found that a very short term (3.6 ks) exposure to 873 K causes the extreme loss of impact toughness before the σ phase formation and also have observed the localized formation of the R phase in the specimen aged for 3.6 ks. Accordingly, the present research concludes that the degradation of the toughness at the early stage of aging (3.6 ks) is attributed to the R phase formation.

2. Experimental Procedures

The chemical composition of the material used is given

in **Table 1**. This alloy was melted in a vacuum induction furnace. After hot rolling, specimens were solution treated at 1323 K for 3.6 ks. Aging treatment was carried out at 873 K for 1.0 to 3.6×10^3 ks followed by water quenching.

Vickers hardness (Load: 98N) measurement and Charpy impact test were performed after the aging treatment. The Charpy impact test was carried out at 273 K and specimen was a half-size one ($5.0 \times 10.0 \times 55.0$ mm V notch) of JIS No. 4 specimen. The microstructural characteristics of the material after the aging treatment were observed by optical and transmission electron microscopes.

3. Experimental Results and Discussions

The Vickers hardness change during the aging treatment at 873 K is shown in **Fig. 1**. The hardness increases with aging time after some incubation period. The results of Charpy impact test are shown in **Fig. 2**. The toughness decreases remarkably after the aging for 3.6 ks and a small peak is observed at about 40 ks aging. We will discuss the relationship between these mechanical properties changes and microstructural evolution during the aging treatment.

Table 1. Chemical composition of the material used. (mass%)

Alloys	C	Si	Mn	P	S	Ni	Cr	Mo	W	Cu	N
25-7-3DSS	0.017	0.48	0.85	0.025	0.001	6.82	24.66	2.79	0.28	0.46	0.143

Fig. 1. Vickers hardness change during the aging treatment at 873 K.

Fig. 2. Charpy impact value during the aging treatment.

Fig. 3. Optical micrographs after (a) solution treatment at 1323 K and aging treatment for (b) 1.0 ks, (c) 3.6 ks, (d) 36 ks and (e) 360 ks at 873 K.

Area A: darkly-etched region in α phase, area B: non-etched region in α phase.

Fig. 4. α and γ phases after the aging for 3.6 ks.

Figure 3 shows optical micrographs after the solution and aging treatment. Island-like γ phase exists in the matrix of α phase. Non-etched α and γ phases after the solution treatment and at 1.02 ks aging are observed in the Figs. 3a and 3b, respectively. Figure 3c indicates that darkly-etched α phase region and non-etched region exist locally after the aging for 3.6 ks, as shown in the areas A and B, respectively. This means that the localized precipitates of second phase exist in the darkly-etched α phase regions. Figures 3d and 3e show that the α phase is uniformly etched, which means the uniform precipitation of some phases in the α phase. The results of the

optical-microscope observation shown in Fig. 3 were investigated in details by the transmission electron microscope (TEM). Figures 4 to 7 are the results of TEM observation. Figure 4 indicates that no precipitate is formed in the α and γ phases and the α - γ interface, after the aging for 3.6 ks. On the other hand, Fig. 5 indicates that, after the same aging treatment, the large precipitates of R phase are formed locally. It is considered that this difference between the precipitation behaviors shown in Figs. 4 and 5 corresponds to the difference between the etching behaviors of the α phase shown in the areas A and B in Fig. 3c, that is, the areas A (etched) and B (non-etched) indicated in Fig. 3c correspond to Figs. 5 and 4, respectively.

After the further aging treatment for 36 ks, a small amount of σ and γ phases is formed as shown in Figs. 6a and 6b. In the Fig. 6b, Widmanstätten-like γ phase precipitates are observed in the α phase matrix. Large precipitates of σ phase are formed at the aging for 360 ks as shown in Fig. 7.

These microstructural observation results indicate that Vickers hardness increases gradually with the formation of small particles of σ phase after the incubation period of about 36 ks.

However, the period of rapid decrease of the Charpy impact toughness does not correspond to that of the gradual increase of the Vickers hardness. This means

Fig. 5. R phase formed after the aging for 3.6 ks
(a) bright field image, (b) dark field image obtained by $(\bar{4}10)_R$ spot, (c) diffraction pattern of R phase and (d) indices of the diffraction pattern of (c).

Fig. 6. σ and γ phases formation after the aging for 36 ks.
(a) σ phase formed at a triple point of grain boundaries (α - α - γ), (b) Widmanstätten-like γ phase formed in an α phase.

Fig. 7. σ phase formed after the aging for 360 ks.

that the mechanisms of the rapid decrease of the Charpy impact toughness and the gradual increase of the Vickers hardness are different. On the Charpy impact toughness, many researchers¹⁻⁶⁾ have reported that the significant decrease of toughness after the aging treatment is attributed to the formation of σ phase. This may be correct, but the rapid decrease of the Charpy impact

toughness cannot be understood by this explanation. Nilsson *et al.*⁷⁾ observed by TEM that R, π and σ phases form after the aging at 873 K for 86.4 ks and that the kinetics of R phase formation is faster than that of the σ phase in a material of 22%Cr-3%Mo-8%Ni (C: 0.012%, Si: 0.47, Mn: 1.61, P: 0.014, S: 0.0009, Cr: 22.32, Mo: 2.95, Ni: 8.19, B: 0.0021, N: 0.15). They also recognized that the Charpy impact toughness decreased significantly after the aging at 873 K for 11.9 ks. From these results, they estimated that the R phase caused the significant decrease of the toughness after the aging at 873 K for 11.9 ks. In the present research, we have made the direct observation of the localized formation of the R phase after the very short time (3.6 ks) aging at 873 K, by TEM, as shown in Fig. 5, while σ phase is not yet formed. Accordingly, the significant decrease of the toughness after the very short time aging is attributed to the formation of the R phase.

Figure 2 showed a small peak of Charpy impact value at the aging time of about 40 ks. This small peak can be attributed to the formation of the Widmanstätten-like γ phase precipitates shown in Fig. 6b, which are con-

sidered to have a role of resisting barrier against the propagation of cracks in the α phase.

4. Conclusions

Microstructural evolution and mechanical properties change during the aging treatment of a 25%Cr–7%Ni–3%Mo duplex stainless steel were investigated.

At the very short-term aging for 3.6 ks at 873 K, the R phase was formed locally, while the σ phase was not formed. This very short-term aging also caused the extreme loss of Charpy impact toughness. From these results, it is concluded that the extreme loss of the toughness due to the R phase formation occurs at the early stage of aging before the embrittlement due to the formation of the σ phase.

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