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Diffraction Study on Aging Duplex Stainless Steel

Toru ISHIGAKI, Takayuki MIURA, Syuuki TORII[1], Takashi KAMIYAMA[2] and Hisashi KUWANO

Department of Materials Science and Engineering, Muroran Institute for Technology, Muroran 050-8585, Japan
[1]*Institute of Material Science, University of Tsukuba, tsukuba, Ibaragi 305-8573, Japan*
[2]*Neutron Science Laboratory, KEK, tsukuba, Ibaragi 305-0801, Japan*

SUS329J2L duplex stainless steels exhibit high strength and resistance to corrosion, so widely used in piping of industrial plants. However, it is known that they are brought deterioration of strength using for long time. This reason of this deterioration is that ferrite decomposes to Fe-rich α phase and Cr-rich α' phase and, Cr-rich α' phase decreases mechanical properties and resistance for corrosion. In this experiment, we made neutron diffraction experiments on long time aging (625 K, 16000 h) duplex stainless steel to observe the behavior for α phase and α' phase, using *Sirius* diffractometer at KENS. The result shows, the lattice parameters in α phase were decreased. In contrast to, its in austenite (γ phase) were slightly increased.

KEYWORDS: SUS329J2L, duplex stainless steels, diffraction study, phase decomposition

§1. Introduction

Duplex Stainless steels are usually used in the oil and power industries at the intermediate temperature range 550 $\sim$ 720 K. However when they are used very long times at the temperature, they can suffer a loss of toughness and corrosivity.[1)] The phase decomposition of ferrite into the Fe-rich α and the Cr-rich α' phase is considered as a main cause of embrittlement,[2)] while the α phase is associated with the loss of corrosion. We have been investigating in this phase decomposition by Mössbauer spectroscopy.[3)] In the present study, the phase decomposition of SUS329J2L duplex stainless steel was investigated by neutron diffraction.

§2. Experimental

A commercial duplex stainless steel, SUS329J2L specification whose chemical compositions are 25%Cr, 7.3%Ni, 3.2%Mo, 0.021%C, 0.52%Si, 0.1%N is solution treated at 1323 K to ensure 65% ferrite. The compositions of ferrite and austenite measured by EDS are shown in Table I. Two plate-like samples (15mm × 65mm × 2mm) were prepared for diffraction experiments. One sample was aged at 623 K for 16000 hours (16000h), and another sample was left as it received for comparison (A.R.). Time-of-flight (TOF) neutron diffraction data were taken at room temperature on the *Sirius* diffractometer[4)] at KENS pulsed neutron scattering facility at High Energy Accelerator Research Organization. The samples were set perpendicular to the incident neutron beam (beam size 10mm × 40mm). Intensity Data was analyzed by Rietveld method for lattice parameters refinement and single-peak fitting.

§3. Result and Discussion

Figure 1 shows the diffraction patterns for a 16000 h aged sample. Lattice parameters of two samples and their difference using neutron diffraction data are listed in Table II.

Table I. Chemical compositions of ferrite and austenite (mass%).

	Cr	Ni	Mo
Ferrite	27.9	5.0	4.3
Austenite	23.8	8.9	2.5

Table II. Lattice parameters (Å) of ferrite and austenite evaluated by Rietveld Analysis.

	AR	16000h	$a_{16000h} - a_{AR}$
Ferrite	2.88005(2)	2.87901(2)	−0.00105
Austenite	3.60492(4)	3.60562(5)	+0.00071

The decrease in lattice parameter of ferrite is well explained by the phase decomposition of ferrite, namely, Cr was replaced by smaller Fe atom in Fe-rich α phase. A compositional change of Fe in the α phase calculated from this decreasing of lattice parameter and the lattice parameter of α-Fe (2.8663Å) and α-Cr (2.8846Å) is abort 6%. This is in good agreement with its calculated by Mössbauer spectroscopy result ($\approx$ 8.5%). On the other hand, we could not find peaks from the Cr-rich α' phase in the diffraction patterns. Mössbauer spectroscopy results showed that the ratio of the α' phase in total sample was only 3.6%,[3)] therefore diffraction peaks from the α' phase seems not to have been observed.

In our previous study using Mössbauer spectroscopy on SUS329J2L, we assumes that the austenite had not change during aging in the temperature range 550 $\sim$ 720 K. On the other hand, Chung *et al.*[5)] reported that the austenite in the CF8M duplex stainless steel (Austenite $\approx$ 80%) have shown decomposed between Fe and Ni. When the decomposition progressed in austenite, Ni was replaced by larger Fe atom at Fe rich phase, *i.e.*, the lattice parameter would become larger. Since, the lattice parameter of austenite in our result was also increased, the austenite in SUS329J2L was also decomposed by long

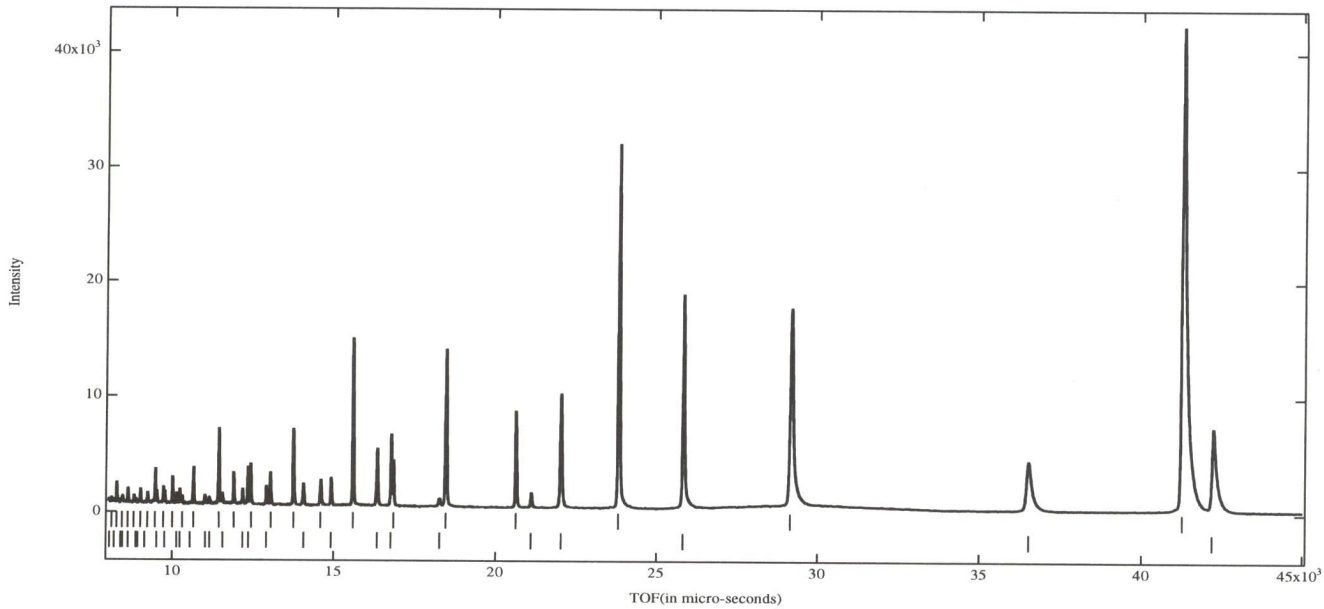

Fig. 1. Neutron diffraction pattern for 16000 h aged sample. Tick marks below the diffraction patterns indicate the positions of possible bragg reflection for ferrite (Upper) and austenite (Lower).

time aging. Only 0.6% of Fe was increased in the austenite that estimated from the change of lattice parameter of the austenite and the lattice parameter of γ-Fe (3.6467Å) and Ni(3.5236Å). The compositional change in the austenite had been too small to observed the phase decomposition using Mössbauer spectroscopy method.

In the lattice parameter refinements, some reflections shifted from their ideal position. We made single-peak fitting to determine the peak positions of each reflections. Figure 2 shows the ratio of peak shift for each reflections between two samples. As shown in the figure, (200) reflection showed the largest change for ferrite. It is consistent with that Fe atoms are concentrate along <100> during decomposition in bcc Fe-Mo alloys.[6] On the other hand, the shift for (400) reflection is not so large, and the shifts of other peak are not change systematically among each reflection. Yamada *et. al.*[3] reported that only 50% of ferrite has been decomposed to Fe-rich α phase at 625 K-16000 h aging process, and the remaining 50% of ferrite are still decomposing. The phase decomposition is not finished at the sample of 16000 h aging also, and the sample contain the fluctuation of concentration between Fe and Cr. This fluctuation of concentration will lead to fluctuation of lattice spacing and various change for peak position of each reflections. In such sample, it may be also caused the peak width broadening. However, the broadening of peak width were not found in Fig 1. To make further discussion about this peak position shifting, more neutron diffraction experiments for the different aging time will be needed.

§4. Conclusions

1. In Ferrite, the diffraction peaks are origin from Fe-rich α phase and the change of Fe concentration (6%) is agree to the result from Mössbauer spectroscopy.

2. In Austenite, we find the existence for the phase decomposition by 623 K 16000 h aging. The change of Fe concentration is 0.6%.

3. It is found that the shift for peak position of each reflection is not systematically. It is need to make further experiments about this.

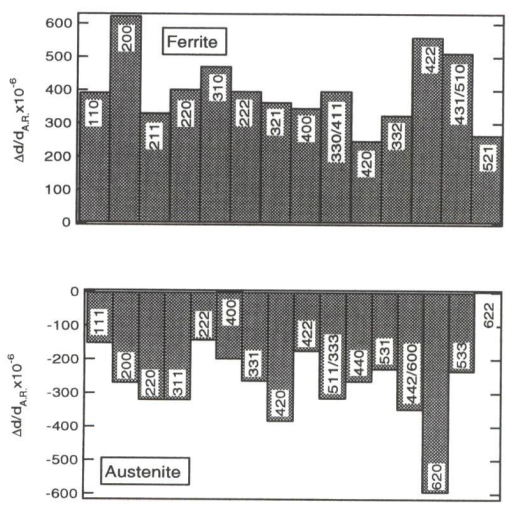

Fig. 2. The changing rate of peak position for ferrite and austenite according aging.

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