Cause of Dynamic Strain Aging in Fe-Cr-Ni Alloys

Sung Soo Kim [a], Dae Whan Kim, Young Suk Kim [a*],
[a]*Hydrogen Cracking Team, Korea Atomic Energy Research Institute, 150, Dukjin-dong, Yuseong, Daejeon 305-353, Republic of Korea*
[*]*Corresponding author: yskim1@kaeri.re.kr*

1. Introduction

Most of the structural materials used in nuclear power plants including the fourth generation reactors are made of Fe-Cr-Ni alloys. For example, Alloy 600 and 316 stainless steel are austenitic alloys containing Fe, Cr and Ni with minor elements such as Mn, Mo or Ti: the former is a nickel base alloy containing 15.5 wt.% Cr and 8wt.% Fe and 1wt.% Mn and the latter is a Fe base alloy containing 12wt.% Ni, 18wt.% Cr and 2 wt.% Mo. One of the mechanical features of these Fe-Cr-Ni alloys is dynamic strain aging (DSA) over a temperature range of 300 to 600^{o}C, whose range strongly depends on the strain rate. As it is well known that DSA lowers fatigue life [1.2], tensile ductility [3] and toughness of the structural components of Fe-Cr-Ni alloys [4], an understanding of DSA is indispensable to mitigating aging of the structural materials used in reactors.

As Fe, Ni and Cr atoms are intermixed as the substitutional elements in the austenitic alloys, the ordered phases of Ni_3Fe [5], Fe_3Ni, Ni_2Cr [6] will be formed only if sufficient thermal and mechanical energies are supplied to the Fe-Cr-Ni alloys. Given that all the ordered phases have low ductility, higher strength and hardness, short range ordering (SRO) to nucleate the ordered phases during tensile tests or fatigue tests will make the Fe-Cr-Ni alloys harder and brittle. Considering that SRO accompanies diffusion of these atoms in the austenitic alloy, the lower strain rate provides more time for diffusion of atoms required to nucleate SRO. Thus, the lower the strain rate becomes, the higher the magnitude of SRO, causing the austenitic Fe-Cr-Ni alloys harder, leading to dynamic strain aging.

The aim of this work is to understand the cause of DSA in 316L stainless steel containing nitrogen concentration varying from 0.01 to 0.15 which is a kind of Fe-Ni-Cr alloys. To this end, the magnitude of stain hardening in the 316L stainless steel was evaluated as a function of strain rate and nitrogen through tensile tests that were conducted with the strain rates changing from $1x10^{-4}$/s to $1x10^{-2}$/s. Evidence for the operation of SRO in 316L stainless steels was provided by determining the lattice contraction induced by SRO with aging time of both a 316L stainless steel and the ordered phases of Ni_2Cr and Fe_3Ni using neutron diffraction. The latter were intentionally made by vacuum induction melting.

2. Experimental Procedures

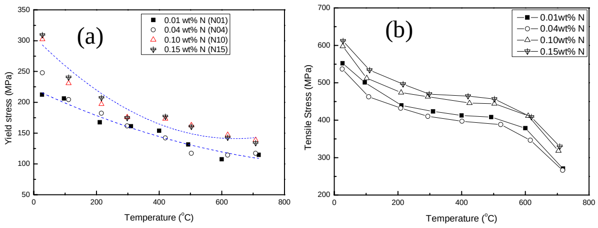

Fig.1. Temperature dependences of (a) the yield and (b) tensile stresses of the 316L stainless steel with nitrogen concentration.

The ingots of 316L stainless steel with nitrogen concentration changing from 0.01 to 0.15 were made in the laboratory by vacuum induction melting. The chemical compositions are given in [1]. They were solution-annealed at 110^{o}C for 1h and water quenched. Tensile specimens were a cylinder shape with a 4mm diameter and 25mm gauge length. Tensile tests were conducted in a temperature range of RT to 700^{o}C with the strain rate changing from $1x10^{-4}$/s to $1x10^{-2}$/s.

The experimental alloys with Ni-34.6 at.% Cr and Fe-25 at.% Ni were made by vacuum induction melting, hot rolled and then aged at 474^{o}C for long time of up to 80,000 h. To provide definitive evidence for SRO, a change of the lattice spacing in 316L stainless steel and the ordered alloys of Ni_2Cr and Fe_3Ni was determined using a neutron diffractometer in Hanaro with aging time. It should be noted that SRO induces a contraction of the lattice spacing, causing a peak shift of the lattice planes to a higher angle. The specific heats of the 316 stainless steel and the ordered phases were determined using a differential scanning calorimeter (DSC) at 10^{o}C/min on heating and on cooling.

3. Results and Discussion

The yield and tensile stresses of a 316L stainless steel containing nitrogen were shown in Fig. 1. A tensile stress plateau was seen to occur from 250 to 500^{o}C but, above 500^{o}C, a rapid drop of the tensile stress occurred independent of the nitrogen concentration. The magnitude of strain hardening termed (TS-YS)/YS decreased with nitrogen concentration and the maximum strain hardening occurred at 600^{o}C for the 316L stainless steel with 0.01wt.% N, which shifted below 600^{o}C at the nitrogen concentrations above 0.01 wt.%. Furthermore, the magnitude of strain hardening was the maximum at 0.01 wt.% N and decreased with increasing nitrogen concentration, as shown in Fig. 2a. Furthermore, a stress increase or $\Delta\sigma$ above the yield stress was the highest at the lowest strain rate as shown in Fig. 2b,

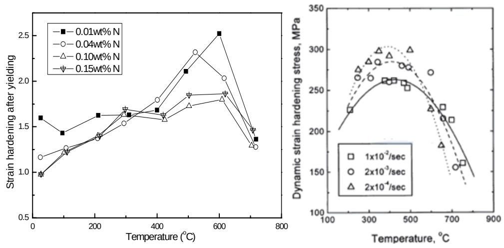

Fig. 2. Strain hardening after yielding of the 316L stainless steel with nitrogen concentration.

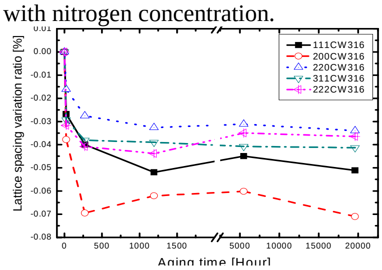

Fig. 3. A change in the lattice spacing of the cold-worked 316L stainless steel with aging time at 400°C.

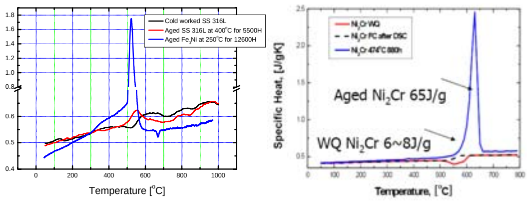

Fig. 4. (a) The specific heats of cold-worked 316L stainless steel and the cold-worked and aged one at 400°C at 5500 h and the aged at 250°C for 5500h and (b) the specific heats of Ni_2Cr displaying the order to disorder transition temperature ranging from 600 to 650°C .

indicating the negative strain rate sensitivity. This fact definitively demonstrates the operation of DSA over a temperature of 300 to 600°C during the tensile tests in the 316L stainless steel.

Given that the ordered phases such as Ni_2Cr, Fe_3Ni and Ni_3Fe are harder than the disordered ones [6], DSA that was seen to occur only at high temperatures under plastic deformation, not under the elastic deformation, is suggested to be due to SRO that nucleates the ordered phases. As ordering contracts the lattice spacing [7], the lattice spacing of the 316L stainless steels that was cold worked to 40% was determined with aging time at 400°C as experimental evidence of SRO. As shown in Fig. 3, the lattice contraction rapidly occurred from 0 to 250h and then leveled off to a constant value for all the planes in the cold-worked 316L stainless steel. This is definitive evidence to demonstrate the operation of SRO in 316L stainless steel. Note that prior cold working of 40% was given to simulate the effect of plastic deformation on SRO at high temperatures during tensile or fatigue tests.

The specific heats of the cold-worked 316L stainless steel and the cold-worked and aged one at 400°C for 5500 h were measured using a differential scanning calorimeter to determine the order to disorder transition temperature. As shown in Fig. 4, the order to disorder transition temperature that was a bit lower for the cold-worked and aged 316L stainless steel as compared to that for the cold-worked one was found to be in the range of 500 to 600°C. In other words, above 600°C, no ordered phases are present due to full disordering. This fact explains disappearance of the tensile stress plateau above 600°C as shown in Fig. 1. Considering the specific heats of the Fe_3Ni and Ni_2Cr, as shown in Fig. 4 and the order to disorder transition temperature of Ni_3Fe of around 510°C, all the three ordered phases are expected to be present below 500°C, the two phases of Fe_3Ni and Ni_2Cr between 500 to 550°C and Ni_2Cr only persists at temperatures between 550 to 600°C. The presence of different ordered phases in 316L stainless steel with temperature is suggested to be the cause of different types of serrations. Evidence for this suggestion is provided by Besag and Smallman [8] who have demonstrated striking yield drops only in the ordered Ni_3Fe, not the disorder one.

4. Conclusions

The 316L stainless steel showed a plateau of the tensile stress, not the yield stress, 4 different types of serrations and a negative strain rate sensitivity over a temperature region of 300 to 600°C, demonstrating the operation of DSA over this temperature region. Nitrogen was found to suppress the DSA. Given that short range ordering to nucleate the hard phases of Ni_2Fe, Fe_3Ni and Ni_3Fe results in hardening, it is suggested that SRO is the cause of DSA in Fe-Cr-Ni alloys including 316L stainless steels. Evidence for the operation of SRO in Fe-Cr-Ni alloys is provided by the lattice contraction of the cold-worked 316L stainless steel with aging time at 400°C. By comparing the specific heats of the 316L stainless steel the ordered phases, it is suggested that nucleation of different ordered phases in 316L stainless steel with temperature is the cause of different types of serrations. Evidence is provided by citing Besag and Smallman's experiment.

ACKNOWLEDGMENTS

This work has been carried out as part of the Nuclear R&D program supported by the Ministry of Knowledge Economy, Korea.

REFERENCES

[1] D.W. Kim, W.G. Kim, W.S. Ryu, Int. J. Fatigue, vol. 25, p.1203, 2003.
[2] S.G. Hong, K.O. Lee, S.B. Lee, Int. J. Fatigue, vo. 27, p. 1420, 2005.
[3] D.W. Kim, W.S. Ryu, J.H. Hong, S.K. Choi, J. Mater. Sci. vol. 33, p. 675, 1998.
[4] S. Iida, J. Phy. Soc. Japan, vol. 7, p. 373, 1952.
[6] A. Marucco, Mater. Sci. Eng. A, vol. 189, p. 267, 1994.
[7] A. Marucco, B. Nath, J. Mater. Sci. vol. 23, p. 2107, 1988.
[8] F.M.C. Begag, R.E. Smallman, Acta Metall. Vol. 18, p. 429, 1970.