Detection of Low Cycle Fatigue in Type 316 Stainless Steel using HTS-SQUID

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A portable RF HTS SQUID-based susceptometer was applied to the measurement of fatigue behavior for type 316L(N) stainless steel containing 0.04% to 0.15% nitrogen content. Strain-controlled low cycle fatigue (LCF) tests were conducted at RT and 600 ℃ in air an atmosphere, and the magnetic moments were measured after the fatigue test using HTS SQUID. The magnetic moment of an as-received sample is higher than that of a fatigued sample in all the temperature ranges irrespective of the nitrogen content. The fatigue life decreased with an increasing test temperature up to 500 ℃, but increased at 600 ℃. The change of the magnetic moments by LCF test is attributed to the stress induced micro defects.

Keywords : *type 316 steel, low cycle fatigue, HTS SQUID, magnetic moment, susceptometer*

1. Introduction

Magnetic techniques have been shown to be useful for crack detection in steel and to be sensitive to the microstructure changes induced by cyclic stress. A SQUID (Superconducting QUantum Interference Device) is the most sensitive magnetic field sensor, with a resolution of 10^{-14} T (tesla) in the 1 Hz bandwidth.[1)] After a relatively general-purpose SQUID-based susceptometer was developed for a wide range of temperature measurements, it was applied in the fields of biomagnetism and geophysical measurement.[2)] In recent years, the SQUID sensor has formed the basis of several new techniques for magnetic nondestructive evaluation (NDE) including the detection of defects in carbon steels, and a system for the detection of damage in materials.[3)] Since SQUID sensors need to be cooled by liquid helium, the equipment is very large and heavy and therefore immobile. Also, the use of liquid helium as a cooling agent restricts a broad application of this type of magnetometer because of its high cost and costly servicing. A recently developed high temperature superconductivity (HTS) SQUID device using liquid nitrogen as a cooling agent was suit to the NDE because of its low cost and relative convenience.[4)] Type 316 stainless steel is a prospective structural material for a liquid metal reactor (LMR) because nitrogen is known to be beneficial to low cycle fatigue (LCF) resistance and decreases the precipitation of carbides.[5),6)] The LMR operating temperature is the most active temperature for dynamic strain aging (DSA) in type 316 stainless steel. The role of DSA and LCF life and the effect of nitrogen on DSA and LCF life has been investigated to apply type 316 stainless steel to high temperature structural components of LMR reactor vessel. The nondestructive detection and evaluation of these cracks are highly desirable from the viewpoint of safe and economic operation of a nuclear reactor. In this study, we have applied the HTS SQUID system to detect the LCF damage for measuring the magnetic properties of type 316 stainless steel with a low magnetic susceptibility.

2. Experimental

2.1 HTS SQUIS system

The SQUID sensor is made of bulk polycrystalline $YBa_2Cu_3O_{7-x}$ ceramics, sensitive to axial (B_z). The interferometer has an axial cylindrical hole of 0.8 mm in diameter and 5 mm high. Its inductance is 1.25×10^{-10} H. Schematic diagram for the HTS SQUID susceptometer is shown in Fig. 1. The resonant tank circuit consists of a coil of L_T ≈» 6×10^{-7} H inductance and a capacitor of C_T ≈» 70 pF, resulting in a quality factor Q ≈» 100. The SQUID is operated at a radio frequency (RF) with a flux bias of 20 MHz. The SQUID flux-to-voltage transfer coefficient reaches a value of 3×10^{10} V/Wb. The system sensitivity is determined mainly by the intrinsic noise characteristic of the sensor, measured within the HTS shield. A HTS RF SQUID-based magnetometer for in

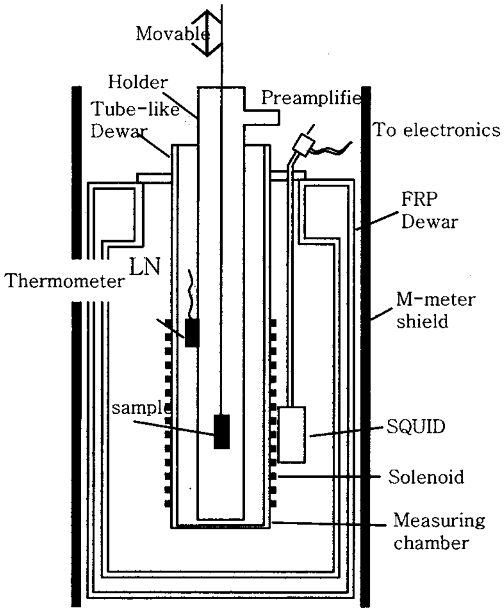

Fig. 1. Construction of HTS RF SQUID-based Susceptometer.

vestigating the magnetic properties of small samples (up to 1.5 mm in diameter) in the weak DC and AC magnetic fields was designed, constructed and tested. The measuring chamber temperature, and therefore the sample temperature, was controlled in the range from 77 ° K up to 150 ° K (and above) by filling the thermo-exchanger with LN or by flowing heated nitrogen gas through the thermo-exchanger.

2.2 Specimen and fatigue test

Four 120 mm thick laboratory ingots, weighing 30 kg each, containing different nitrogen contents were prepared by vacuum induction melting (VIM). The chemical compositions of the ingots are listed in Table 1. Fatigue tests employed a round bar specimen with a 7 mm diameter and 8 mm gauge length. The samples for the SQUID test were extracted from the position of the grip and gauge section as shown in Fig. 2. All the specimens were held at the test temperature for 1 h before the test were started. The temperature was maintained to with in 2 K during the period of the test. The experiments were conducted

Table 1. Chemical composition of specimens (wt%)

Spec. ID	C	Si	Mn	Ni	Cr	Mo	N	P (max.)	S (max.)
N04	0.018	0.67	0.95	12.21	17.78	2.36	0.042	0.007	0.002
N10	0.019	0.70	0.97	12.46	17.23	2.38	0.103	0.007	0.002
N12	0.019	0.70	0.96	12.45	17.17	2.39	0.121	0.007	0.002
N15	0.020	0.67	0.96	12.19	17.88	2.41	0.151	0.007	0.002

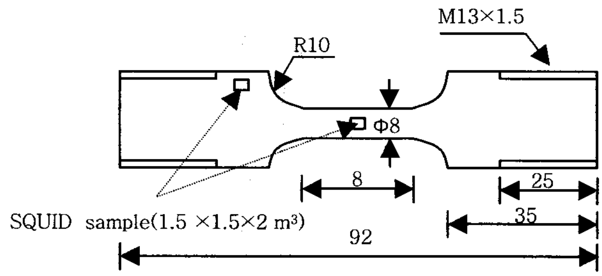

Fig. 2. Configuration of fatigue specimen and extraction position of HTS SQUID sample.

at a strain rate of 1 x 10^{-3} s^{-1} and from room temperature (RT) to 600 ℃. The SQUID is operated at a radio frequency (RF) with a flux bias of 20 MHz.

3. Results and discussion

The change of the magnetic moment of the as-received and fatigued samples with nitrogen contents is shown in Fig. 3. The magnetic moment of the as-received sample is higher than that of the fatigued sample in all the temperature ranges irrespective of the nitrogen contents.

Defects in materials such as microstructure and micro crack by cyclic stress during the fatigue process make magnetization more difficult.[7] Therefore the hysteresis loop of the deformed sample is lower than that for the fully annealed condition. The resulting micro stress impedes both the domain wall motion and domain rotation, decreasing the magnetic moment. In case of type 316 stainless steel, many factors influence the motion of domain wall including micro crack, and various dislocations having a planar and cell structure. The reason that the magnetic moment of the fatigued sample is lower than that of the as-received one is attributed to these defects.

The magnetic moments of the as-received sample gradually decrease with an increasing nitrogen content. The magnetic moment of the as-received sample having a nitrogen content of 0.04 wt is about two times that of 1.5 wt. On the other hand, the magnetic moment of the fatigued sample shows little change with an increasing nitrogen content, but it shows slightly higher and lower values in the sample having a nitrogen content of 0.1 wt and 0.12 wt, respectively.

It is known that nitrogen decreases magnetic moment by stablizing the austenitic phase.[8] A low magnetic moment of stainless steels is linked to the stability of their austenitic structure. The more stable the austenitic structure is, the lower the magnetic moment of the stainless steel is. Therefore, a low magnetic moment in the as-received sample with a high nitrogen content seems to

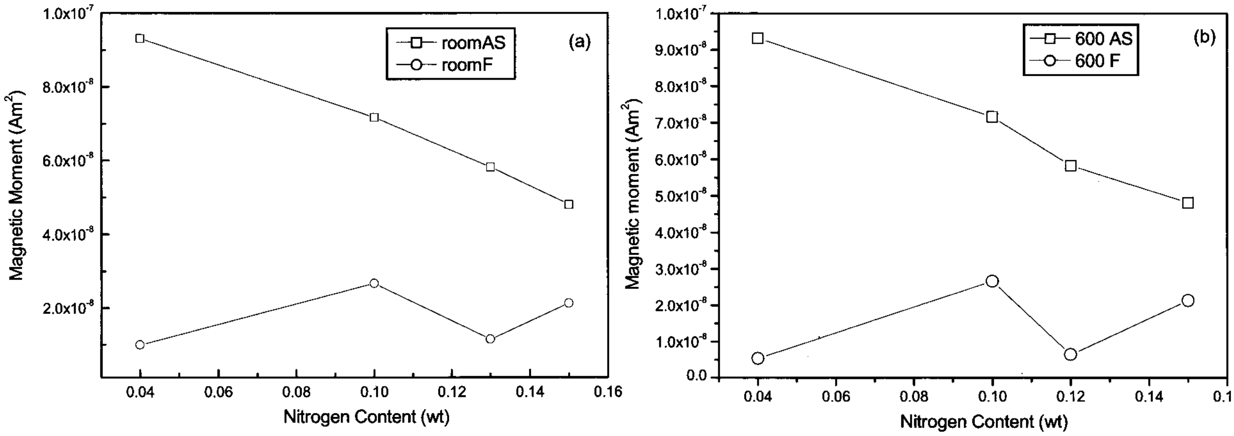

Fig. 3. Change of magnetic moment with nitrogen content (a) tested at room temperature (b) tested at 600 ℃

be attributed to the nitrogen induced austenitic phase. Kim et al. reported that strain induced martensite decreases with the addition of nitrogen at RT.[9] However, the change of the magnetic moment with the nitrogen contents in the fatigued sample could not be explained by the strain induced martensite because the lower martensite phase is expected to decrease the magnetic moment which is contrary to our results. It is known that martensite transformation can take place near a fatigue crack edge as a result of cyclic softening and hardening during the fatigue process in the 316 stainless steel. In the vicinity of the fatigue crack edge, the martensitic transformed phase is produced uniformly in each grain, and around this region the martensitic transformed phase is produced selectively in specific grains or in a specific region in a grain.[11] The increase of magnetic moment in the fatigued sample could

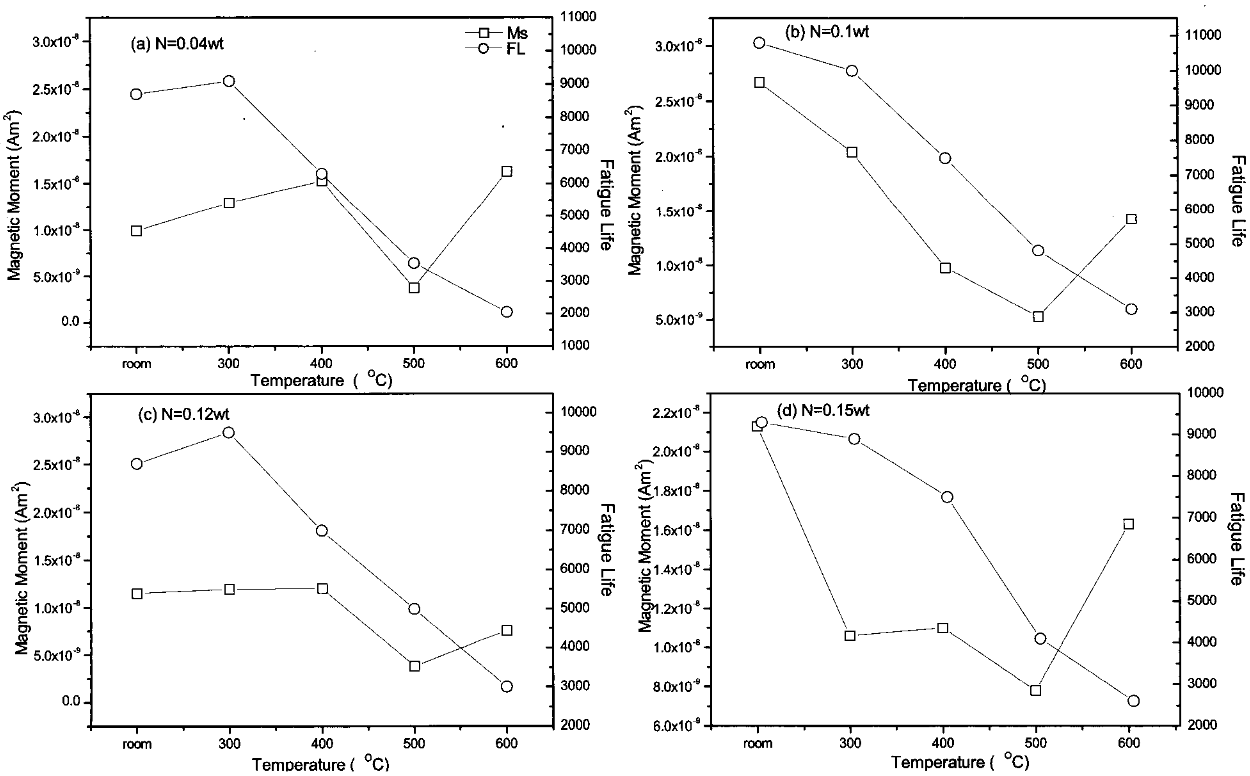

Fig. 4. Temperature dependence of magnetic moment and fatigue life with nitrogen contents.
(a) nitrogen content of 0.04wt, (b) 0.1 wt, (c) 0.12 wt, (d) 0.15 wt.

be a result of the creation of the martensitic transformed phase around fatigue edge.

Fig. 4. shows the temperature dependency of the magnetic moment and fatigue life with the nitrogen contents. The fatigue life decreases with an increasing test temperature in all the samples, but the magnetic moments decrease gradually up to the test temperature of 500 ℃ and increase at 600 ℃. It is known that crack propagation rate increase with the test temperature and decrease with the nitrogen content.[9] Therefore, the decrease of fatigue life with increase test temperature is attributed to the increase of crack propagation rate. The decrease of magnetic moment up to the test temperature 500 ℃ is attributed to the increase of the crack propagation. It is known that nitrogen changes the dislocation structure from a cellular to a planar, decreases the grain size, reduces the strain induced martensite, and retards DSA. Kim et al. reported that a larger decrease of fatigue life at 600 ℃ than that observed at RT is attributed to the retardation of DSA associated with the addition of nitrogen.[10] Retardation of DSA can be accompanied with an elimination of a micro crack associated with the dislocation structure, which results in an increase of the magnetic moment. Therefore, the increase of magnetic moment in the 600 ℃ fatigued sample is attributed to the elimination of the micro crack.

4. Conclusion

The SQUID measurements for the strain controlled LCF samples were conducted. The reason that the magnetic moment of the fatigued sample is lower than that of the as-received one is attributed to the stress induced defects. The decrease of magnetic moment in the as-received sample with a high nitrogen content seems to be attributed to the nitrogen induced austenitic phase. The increase of the magnetic moment in the fatigued sample containing high nitrogen is a result of the cyclic softening. The decrease of fatigue life with increasing test temperature is attributed to the increase of crack propagation rate. The decrease of magnetic moment up to the test temperature 500 ℃ is attributed to the increase of the crack propagation. The increase of the magnetic moment in the 600 ℃ fatigued sample is attributed to the elimination of the micro crack.

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