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ScienceDirect
Journal of Magnetism and Magnetic Materials 320 (2008) e571–e574
Journal of magnetism and magnetic materials
www.elsevier.com/locate/jmmm

Measurement of the magnetic moment in a cold worked 304 stainless steel using HTS SQUID

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Available online 10 April 2008

Abstract

The magnetic properties of stainless steel have been investigated using a radio frequency (RF) high-temperature superconductivity (HTS) SQUID (Superconducting QUantum Interference Device)-based susceptometer. The nuclear grade 304 stainless steel is nonmagnetic at a normal condition but it changes to a partially ferromagnetic state associated with martensitic transformation under a plastic deformation. The magnetic moment of the 304 stainless steels was increased with an increasing cold work rate, and decreased with an increasing annealing temperature. The change of mechanical properties such as yield strength and ultimate tensile strength (UTS) are also analyzed in terms of deformation-induced martensitic transformation.

PACS: 62.20.M; 85.25.Dq; 85.25.Cp

Keywords: HTS SQUID; Stainless steel; Martensitic transformation; Cold work; Yield stress

1. Introduction

SQUID (Superconducting QUantum Interference Device) has the potential for a nondestructive evaluation (NDE) of a degradation or nano-size crack in the pipeline of nuclear power plants (NPPs). A SQUID is the most sensitive magnetic field sensor, with a resolution of 10^{-14} T (tesla) in the 1 Hz bandwidth region [1]. In recent years, the SQUID sensor has formed the basis of several new techniques for a magnetic NDE including the detection of defects in carbon steels, and a system for the detection of damage in materials [2]. Since SQUID sensors need to be cooled by liquid helium, the resulting 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 found to be more suitable for an NDE because of its low cost and relative convenience [3]. On the other hand, in order to prevent serious accidents in the pipeline of a NPP, it is very important to estimate the amount of fatigue damage in the austenitic stainless steel (SS), which is widely used as pipeline materials for a NPP [4]. If we can estimate the amount of damage in the pipeline, the service life of a structure can be extended and an accident can be prevented. Recently studied SQUID-based magnetometers are effective for estimating the amount of fatigue damage of an austenitic pipe in a NPP. It is well known that a transformation from an austenite to a martensite is induced by a plastic deformation [5]. Most previous reports refer to the response of these evaluations in terms of the effects of the martensite contents in 304 SS with a progressive cold rolling [6,7]. However, it is a fact that many of these structures experience a unidirectional tensile loading, which incurs a lot of deformation induced by a martensite generation and growth. Type 304 SS, which is used in the pipeline materials of NPP, is nonmagnetic under normal conditions, but changes to a ferromagnetic state by the martensitic transformation under a plastic deformation such as fatigue damage. The present investigation was aimed at NDE of 304 SS during deformation. The study

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0304-8853/$ - see front matter © 2008 Published by Elsevier B.V.
doi:10.1016/j.jmmm.2008.04.037

has been carried out on its virgin sample, which is nonmagnetic and also on cold-rolled materials containing low volume fraction of α martensite, which is ferromagnetic.

2. Experimental

2.1. HTS SQUID 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 it is 5 mm high. Its inductance is 1.25×10^{-10} H. A schematic diagram of the HTS SQUID susceptometer is shown in Fig. 1(a) and (b). The amplitude and phase of this compensation current can be finely adjusted in order to minimize the output signal, ideally to zero. When the tested sample is moved inside the solenoid near a SQUID sensor, the compensation is discontinued. The amplitude of the output signal is proportional to the sample DC or AC magnetization. The resonant tank circuit consists of a coil of a $L_T \approx \geqslant 6 \times 10^{-7}$ H inductance and a capacitor of $C_T \approx \geqslant 70$ pF, resulting in a quality factor of $Q \approx \geqslant 100$. 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's sensitivity is determined mainly by the intrinsic noise characteristic of a sensor, measured within an HTS shield. A HTS RF SQUID-based magnetometer for investigating the magnetic properties of small samples (up to 3 mm in diameter) in 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 up to 150 K (and above) by filling the thermo-exchanger with LN or by flowing heated nitrogen gas through the thermo-exchanger.

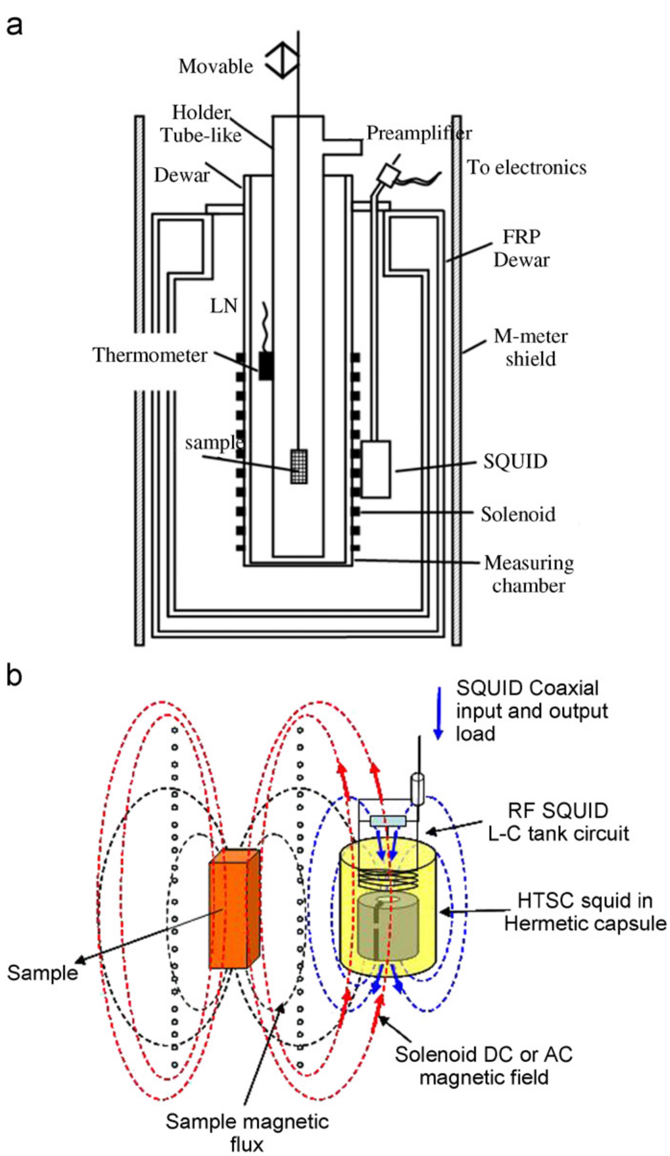

Fig. 1. Construction (a) and measuring principle (b) of the HTS RF SQUID-based susceptometer.

2.2. Specimen and fatigue test

The material used for the investigation was 304 SS. The chemical composition of the material is given in Table 1. Specimens were first solution annealed in a vacuum furnace at 1100 °C for 1 h and then water quenched. Tensile test was employed to 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. The temperature was maintained to within 2 K during the period of the test. All the specimens were held at the test temperature for 1 h

Table 1
Chemical composition of the solution annealed and cold worked type 316 SS (wt%)

Spec.	C	Si	Mn	P	S
SA304	0.045	0.406	1.688	0.03	0.001
CW304	0.034	0.47	1.13	0.029	0.0036
Spec.	Cr	Ni	Mo	Cu	N
SA304	18.23	8.155	–	–	0.062
CW304	18.38	9.08	0.29	0.44	0.026

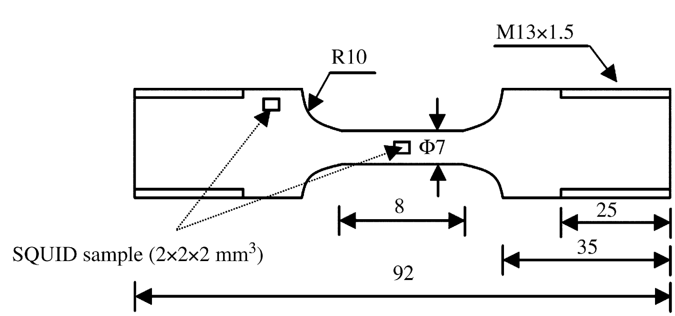

Fig. 2. Configuration of the fatigue specimen and the extraction position of the HTS SQUID sample.

before the tests were started. The experiments were conducted at a strain rate of $1\times10^{-3}\,s^{-1}$ and from room temperature (RT) to 600 °C.

3. Results and discussion

Fig. 3(a) and (b) shows the variation of the magnetic moment for the solution annealed and cold worked samples, which were tested at various cold work rates and heat treatment temperatures. The magnetic moments of the sample tested at room temperature increased abruptly with an increasing cold work rate, but that of the samples tested at high temperature increased slightly with an increasing cold work rate, and that of the 1.5% cold worked sample was greater by more than 10 times than that of the solution annealed sample. The magnetic moment of the solution annealed sample did not change with increasing test temperature, but that of the cold worked sample was considerably decreased with an increasing test temperature. The increase of the magnetic moment in the cold worked sample can be explained by a creation of a ferromagnetic phase by a stress. Nuclear grade 304 SS is nonmagnetic and hence shows almost no response to a magnet when in an annealed condition; however, there can be a magnetic response due to an atomic lattice straining and the formation of a martensite. The austenitic structure of these alloys is metastable and a martensite transformation from (fcc) γ to (bcc) α can occur, producing a ferromagnetic phase, depending on the strain level and temperature [8,9]. The volume fraction of the α martensite depends on the temperature and the amount of a plastic strain. At a temperature above 300 °C much less α martensite is obtained. The specimens can be considered to be nearly paramagnetic at a temperature above 300 °C. The results of the magnetic measurements suggest that a martensitic transformation occurred and below 300 °C the percentage of the α martensite depends on the amount of a plastic strain. The martensitic transformation gradually decreases until no further transformation occurs upon a further straining, and a limiting value for the martenstitic content is reached. The higher the temperature, the lower the vol% of α martensite, until a certain temperature is reached. In general, the higher the nickel to chromium ratio the more stable the austenitic structure is, and the less the magnetic response that will be induced by a cold work. The 304 austenitic SSs, which have a magnetic response due to a cold work, can be returned to a nonmagnetic state by a stress relieving.

Therefore the high magnetic moment in the cold worked sample is attributed to the formation of a stress-induced a martenstite phase, and the low magnetic moment in the samples tested at a high temperature is attributed to a stress relieving due to an annealing.

Fig. 4(a) and (b) shows the tensile properties with the temperature for the solution annealed and cold worked 304SS. Yield stress and ultimate tensile strength (UTS) increased in the cold worked steel for the whole temperature range. The yield stress of the solution annealed steel decreased slowly above 300 °C, but that of the cold worked steel decreased slowly up to 500 °C and rapidly decreased above this temperature. In the case of the yield stress, the effect of a cold working is dominant at a low temperature, but it decreases at a high temperature. Steel with 8–11% Ni, such as 304 SS, becomes harder after a plastic deformation not only as a result of a work hardening but also because of a martensitic transformation induced by a plastic deformation [10]. As a result of a phase transformation induced by a deformation, 304 SS has a certain amount of α (bcc) and ε (hcp) martensite, which is a function of the temperature, alloy composition and plastic

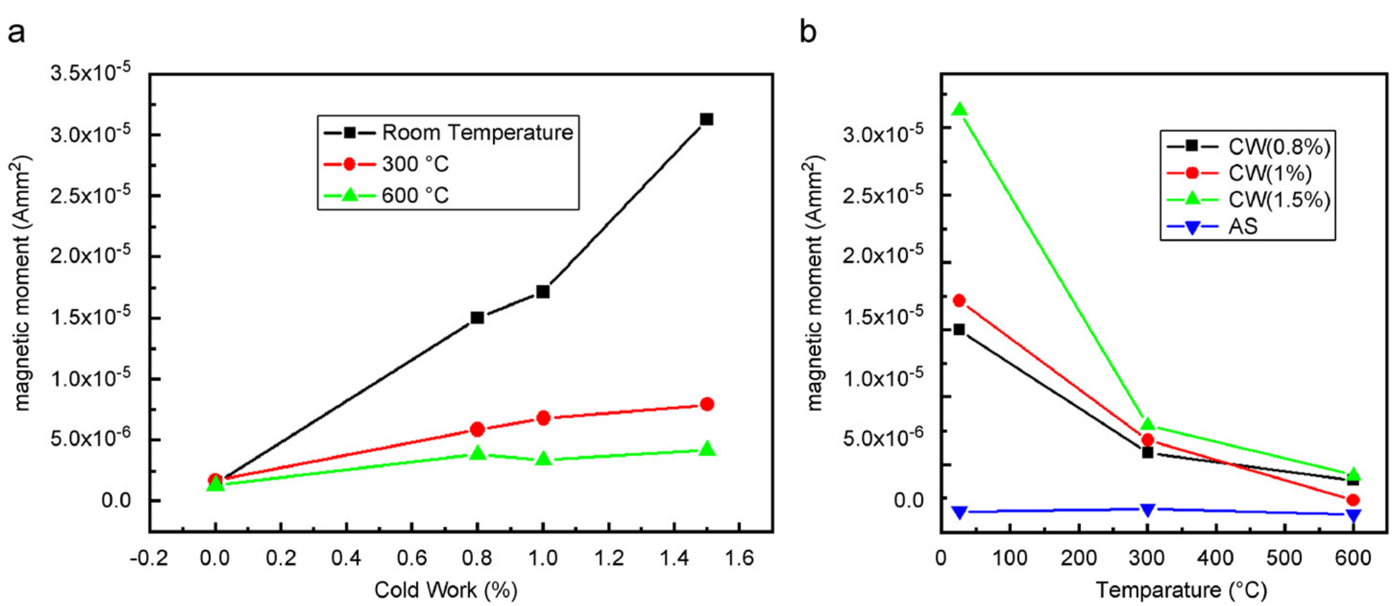

Fig. 3. The change of the magnetic moment (a) as a function of the cold work rate at different heat treatment conditions and (b) as a function of test temperature at different cold work rates.

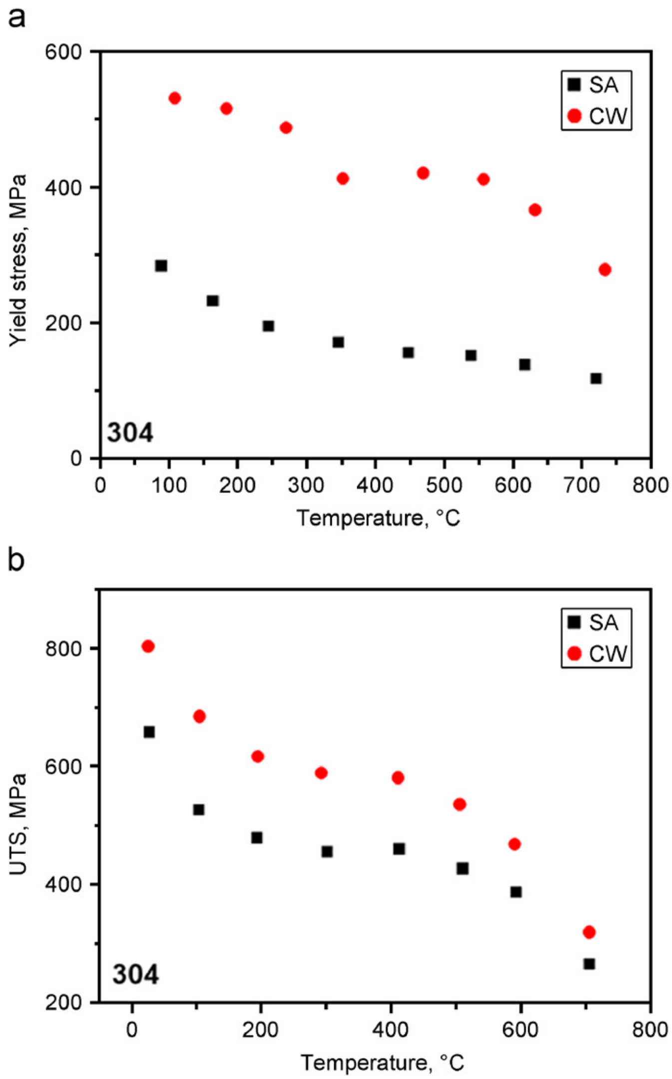

Fig. 4. Tensile properties with temperature for solution annealed and cold worked type 304 SS: (a) yield stress and (b) ultimate tensile strength.

deformation [11]. Experimental results suggest that the martensite phase is responsible for the higher mechanical strength of these samples relative to undeformed samples [12].

Therefore the large difference of the yield stress and UTS between the solution annealed and cold worked steel below 500 °C is attributed to the strain-induced martensitic transformation.

4. Conclusions

The magnetic moment of the 304 SS samples was measured at various cold work rates and heat treatment temperatures using HTS SQUID. The magnetic moments of the samples tested at room temperature increased abruptly with an increasing cold work rate, but that of the cold worked samples increased slightly with an increasing cold work rate, and that of the 1.5% cold worked samples was greater by more than 10 times than that of the as-received sample. The yield strength of the solution annealed steel decreased slowly above 300 °C, but that of the cold worked steel decreased slowly up to 500 °C and rapidly decreased above this temperature. The increase of the magnetic moment in the cold worked samples can be explained by a strain-induced martensite transformation. A large difference of the yield stress and UTS between the solution annealed and cold worked steels below 500 °C is attributed to the strain-induced martensite transformation.

Acknowledgment

This work has been carried out as a part of the advanced diagnostic techniques for micro materials degradation and damage project under the Nuclear R&D Program by MOST in Korea.

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