phys. stat. sol. (c) **4**, No. 12, 4548–4551 (2007) / **DOI** 10.1002/pssc.200777269

New method for detecting the early stage of structural changes by using HTSC squid-based magnetometer

D. G. Park[*,1], **A. Chandrasekhar**[1], **D. W. Kim**[1], **V. P. Timofeev**[2], and **Y. M. Cheong**[1]

[1] Korea Atomic Energy Research Institute, 105 Dukjindong, Yusongku, Taejeon, Korea
[2] Verkin Institute for Low Temperature Physics & Eng. National Academy of Sciences of the Ukraine, 47 Lenin Ave., 61103 Kharkov-103, Ukraine

Received 7 May 2007, revised 16 July 2007, accepted 5 November 2007

PACS 07.55.Ge, 61.66.Dk, 75.60.Ej, 85.25.Dq

The new method of detection the early stage of structural changes in stainless steel samples is proposed and used. It includes the registration of initial magnetization line shift, the zero region of magnetic hysteretic loop (*B-H*). This method is based on the use of high sensitive SQUID magnetic sensor. As a result, the spontaneous magnetic moment that treated sample manifests in zero external magnetic fields is recoded. The magnetic moments of cold worked 316 stainless steel have been measured as an application of this type of HTSC (High Temperapture SuperConducting) SQUID-based magnetometer.

1 Introduction

Materials Non Destructive Evaluation (NDE) is the major concern in both production and maintenance inspection of mechanical parts and systems. Magnetic techniques have been shown to be useful for detection of the microstructure changes and defects of the steel, because magnetic responses give information about structural heterogeneity in the materials [1]. Conventional eddy-current inspection of conducting objects is a set of well established methods that have already proven to give reliable information about cracks, fatigue and corrosion inside workpieces. However, the investigation of deep defects is still a challenging problem. This is because the sensitivity of conventional sensors is rather poor at the low frequencies required to ensure deep penetration of the excitation fields into conducting material. Squid sensors having high and frequency independent sensitivity (down to few parts of Hz) are good for this purpose. Superconducting QUantum Interference Devices (SQUIDs) are the most sensitive magnetic sensors which have a resolution up to few fT (10^{-15} tesla) at a frequencies range from few and even portion of Hz up to few MHz [2]. The use of liquid helium as a cooling agent restricts a broad application of this type of magnetometer because of its high cost and complicated servicing. Liquid nitrogen cooled HTSC squid-based magnetometer shows high sensitivity and require only reasonable expenses to be used and maintained. In this study, we developed this type of magnetometer system, and applied in measuring the sample magnetic moment appeared after cold work in 316 stainless steel which is used in the pipe line materials of nuclear power plant [3].

2 HTSC squid-system and methods

The magnetic hysteretic loop is generated by measuring the magnetic flux of a ferromagnetic material while the magnetizing force is changed. A ferromagnetic material induction B that has never been previ-

---

[*] Corresponding author: e-mail: dgpark@kaeri.re.kr, Phone: +81 42 868 2023, Fax: +82 42 868 8549

ously magnetized or has been thoroughly demagnetized will follow the solid line as H is increased as shown in Fig. 1.

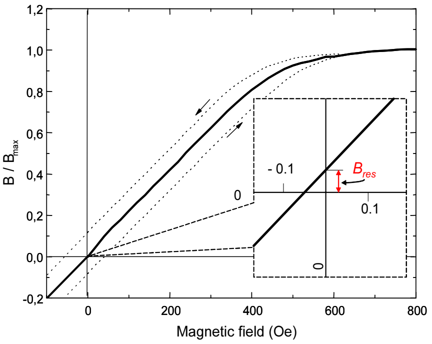

Fig. 1 A portion of hysteresis loop showing the initial magnetization and demagnetization of typical tested samples.

Residual magnetism or residual flux is the magnetic flux density B_{res} that remains in a material when the magnetizing force is zero. In this case, the residual magnetic moment M_{res} and residual magnetic flux density B_{res} is the result of sample after mechanical test. Because of these treatment there appeared martensite phase precipitations and the spontaneous, intrinsic magnetic moment of tested samples. Due to high sensitivity of squid magnetic sensor we can record the initial residual magnetic moment (vertical shift of line part of the steel magnetization) – B_{res} in zero external magnetic field. In HTSC SQUID-based system we used there is high efficient multilayer magnetic shields. The suppression of external constant magnetic fields is about 60 dB (10^3 times). When the system solenoid current is not switched (or turn off) H is really zero (residual magnetic field inside the multilayer magnetic shield is about 0.4 mOe).

2.1 Squid system DC and AC measurement possibility

DC magnetic measurement, when a direct current in solenoid generates constant and uniform magnetic field, gives the equilibrium value of the magnetization and magnetic moment of a sample. In AC magnetic measurements, an alternating magnetic field is applied to tested sample and the resulting AC magnetic moment is measured, are an important tool for material characterization. Because the induced sample magnetic moment is time-dependent, AC records yield information about magnetization dynamics which are not obtained during DC measurements.

When one going to begin DC or AC measurements by SQUID-based system the first needs to avoid direct influence the solenoid magnetic field on sensor. The systems cooled with liquid helium have highly balanced gradiometric pick-up coils made from thin niobium wire and niobium shielded SQUID sensor for this purpose. The result is that the magnetic sensor is not sensitive to uniform DC or AC magnetic fields generated by solenoid, but it is sensitive to magnetic fields caused by the magnetized sample. Unfortunately, there is no suitable HTSC wire cooled with liquid nitrogen till now. Thus it is need to design electronic compensation circuit. We propose one of a possible version for this scheme with minimum additional input leads which are, as a rule, the source of electromagnetic interferences.

AC magnetic susceptibility measurements can provide valuable information about microscopic sample structures, especially when they are frequency dependent as, for example, in ferromagnetic. The SQUID sensor is sensitive to any variation in magnetic field. Therefore, the system design must prevent any effect due to the presence of homogeneous AC magnetic field of the solenoid appearing in the output signal. Because of the absence in practice of HTSC wire for the dB_z/dz pickup coils forming, this task is very difficult using liquid nitrogen cooled system. But for measurement in DC or AC magnetic fields, a special designed electronic circuit can be used for compensation of the output signal in the absence of tested sample (or when the sample is moved off solenoid) (see Fig. 2).

The exciting DC or AC current from DC current source or the standard low frequency generator is passed through the solenoid, and part of this current via tuneable phase shifter is fed to SQUID resonant circuit coil. This current should produce the magnetic flux inside the SQUID interferometer in the opposite direction to the solenoid field. 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 to the SQUID sensor, the compensation is discontinued. The amplitude of the output signal is proportional to the sample DC or AC magnetization.

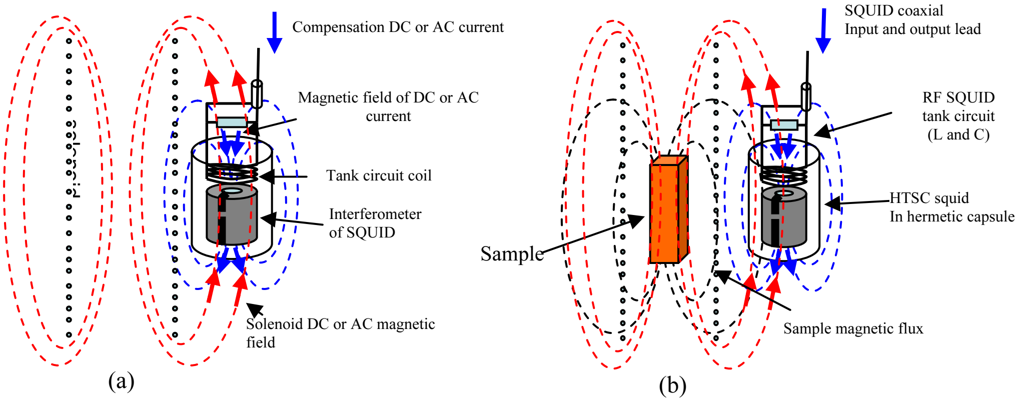

Fig. 2 (a) The amplitude and phase of compensation current can be finely adjusted in order to minimize the output signal, ideally to zero. This means that inside SQUID interferometer magnetic flux generated by solenoid is equal and opposite to magnetic flux produced by compensation current in resonant tank coil. (b) The tested sample is moved inside the solenoid near to SQUID then compensation is discontinued, hence the output signal is appeared.

2.2 Construction of squid sensor

The SQUID sensor has been made of bulk polycrystalline $YBa_2Cu_3O_{7-x}$ ceramics, sensitive to axial (vertical) component of vector B_z and sample magnetization M_z . The interferometer of SQUID has an axial cylindrical hole 0.8 mm in diameter and 5 mm high. Its inductance is 1.25×10^{-10} H. The resonant tank circuit consists of a coil of $L_T \approx 6\times10^{-7}$ H inductance and a capacitor of $C_T \approx 70$ pF, resulting in a quality factor Q ≈» 100. The SQUID is operated with a commonly used 20 MHz radio frequency (RF) pumping. The system sensitivity is determined mainly by the intrinsic noise of the sensor equals to 5×10^{-13} T/Hz$^{-1/2}$, measured within the HTSC shield. But in practice the system sensitivity was limited by local level of external electromagnetic interferences. As a result system resolution to magnetic moment of samples was of the order of 5.8×10^{-10} A·m^2. The HTSC RF squid-based magnetometer for investigating the magnetic properties of small samples 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 liquid nitrogen (LN) or by flowing heated nitrogen gas through the thermo-exchanger.

2.3 Specimen and mechanical test

Nuclear grade 316 stainless steel sample have been used in the SQUID performance test. Cold work tests employed round bar specimen with 7 mm diameter and 8 mm gauge length. All the specimens were held at the test temperature for 1 hour before the test were started. The temperature was maintained to with in 2 K during the period of the test. The experiments were conducted at a strain rate of 1 x 10^{-3} s^{-1} and from room temperature (RT) to 600 °C.

3 Results and discussions

The measurement of magnetic properties were conducted in the 316 stainless steel sample as-received and after cold work conducted at 300 and 600 °C. Figure 3 shows variation of magnetic moment for the as-received sample and cold worked sample which is tested in the various cold work rate and heat treatment temperature. The magnetic moments of as-received sample did not change with heat treatment temperature, but that of cold worked samples decreased with increasing of heat treatment temperature. The magnetic moments tested at room temperature increased with increasing cold work rate, and that of 1.5 % cold worked sample is greater more than 10 times than that of as-received sample. But, the magnetic moment of cold worked sample tested in the high temperature considerably decreased. The increase of magnetic moment in the cold worked sample can be explained as a creation of ferromagnetic phase by stress. Nuclear grade 316 stainless steel is non magnetic and hence show almost no response to a magnet when in the annealed condition, however, there can be a magnetic response due to atomic lattice straining and formation of martensite. In general, the higher the nickel to chromium ratio the more stable is the austenitic structure and the less magnetic response that will be induced by cold work.

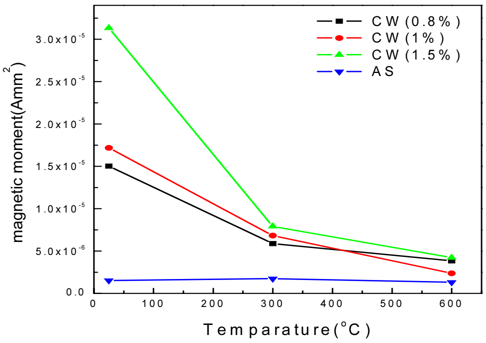

Fig. 3 Dependence of magnetic moment on different heat treatments condition of cold worked samples.

The 316 austenitic stainless steels which have magnetic response due to cold work can be returned to a non-magnetic condition by stress relieving. Therefore high magnetic moment in the cold worked sample is attributed to the formation of martenstitic phase by stress, and low magnetic moment in the samples tested at high temperature is attributed to the stress releving due to annealing [4].

4 Conclusion

The new method of detection the early stage of structural changes in stainless steel samples is proposed and used. This method is based on the use of high sensitive SQUID magnetic sensor. It includes the registration of initial magnetization line shift, the zero region of magnetic hysteretic loop (*B*-*H*) and possibility of combination the DC and AC measurements. As a result, the spontaneous magnetic moment that treated sample manifests in zero external magnetic fields is recoded. A portable HTSC RF SQUID system is developed for the nondestructive evaluation of a stainless steel which is used in the pipeline of nuclear power plant. The performance tests were conducted using cold worked 316 stainless steel. The abrupt increase of magnetic moment in the cold worked sample is attributed to the formation of stress induced martenstitic phase.

References

[1] G. B. Donaldson, phys. stat. sol. (c) **2**, 1463 (2005); H. Weinstock, IEEE Trans. Magnetics **27**, 3231 (1991).
[2] R. Kleiner, D. Koelle, F. Ludwig, and J. Clarke, Proc. IEEE **92**, 1534 (2004).
[3] D. G. Park, D. W. Kim, V. P. Timofeev, and J. H. Hong, Key Eng. Mater. **270-273**, 1224 (2004).
[4] http://www.atlasmetals.com.au/