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A NOVEL VSM MAGNETOMETER AND ITS APPLICATION TO DETECT INDUCED MAGNETIC ANISOTROPY

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Nowadays, there is increasing importance of the remaining life time estimation of these engineering structures. In the present paper we summarize some of our results obtained in the field of magnetic testing of creep tested power plant steel. In this work 15Mo3 type ferritic heat resistant steel was investigated. This steel and several similar grades are commonly used in power plants boilers as the material of reheating steam pipelines and pressure vessels. Their typical application temperature is about 500-550 °C. It is commonly accepted that a combined form of mechanical, thermal fatigue and the creep processes causes the long term deterioration of this structural steels. The applied thermal shock fatigue and creep tests can properly model the material degradation due to long term service in high temperature environment. A parallel motion vibrating sample magnetometer was used for testing the magnetic properties of the samples and several different magnetic properties were measured to characterize the microstructural processes of the deterioration. For detecting the induced magnetic anisotropy due to creep and thermal shock fatigue processes a novel parallel motion vibrating sample magnetometer (VSM) was developed in our department.

Keywords: vibrating sample magnetometer, creep, induced magnetic anisotropy, remnant life

1 INTRODUCTION

The state of power plant steels is thermodynamically metastable and the structure of these materials can change during long term service, especially at elevated temperature. In the real power plant overheating pipelines the most important deterioration processes are the creep and a combined form of mechanical and thermal fatigue due to service temperature cycles. The typical appearance of the macroscopic deterioration of these pipelines is the longitudinal fracturing. It is known that the microstructural effects of the two mentioned processes can be distinguished because the creep type deterioration can cause cavities along the grain boundaries, while the fatigue process causes the appearance of micro and later macro cracks through the grains. During the high-temperature operation the crack growth under the above mentioned complex loads can be classified as cycle- or time-dependent or a combination of the two processes. Cycle-dependent crack growth is due to fatigue and time-dependent crack growth is due to creep process or to environmental interactions. A combination of their effects is also possible [1].

A new type of vibrating sample magnetometer (VSM) was designed for measuring the magnetic properties of soft and hard magnetic materials is described. The developed instrument differs from the traditional Foner type because in our system the motion of the specimen is parallel with the lines of the external magnetic field. Therefore, this instrument can be called parallel motion vibrating sample magnetometer (PMVSM). The special vibrating system contains a vibrating rod which holds the specimen. This arrangement can make the sample replacement and positioning fast and convenient. Because of the versatility of the PMVSM instrument it could be a useful measuring device for materials science laboratory and educational purposes as well.

The vibrating sample magnetometer (VSM) first developed by Foner in the late 1950s [2]. This equipment is really universal because the same measuring arrangement can be used for testing soft and hard magnetic materials. The most important advantages of VSM are the relatively small specimen size, the high applicable external field and the possibility to measure magnetic films as well. The VSM is an open magnetic loop measurement technique therefore, for the determination of the magnetic field strength inside the sample its demagnetisation factor must be considered. The VSM magnetometers could have two more features. The magnetic properties of the specimen can be investigated in different directions therefore the equipment is suitable for studying the magnetic anisotropy.

The basic principle of VSM is, that if any material is placed into magnetic field a dipole moment will be induced in the sample proportional to the product of the sample susceptibility and the applied field. If the sample vibrates sinusoidaly, the resulting magnetic flux changes near the sample will induce an electrical signal in suitable placed detector coils. The induced voltage is proportional to the moment of the sample, the amplitude and frequency of the vibration. Using a vibration controlling reference coil the amplitude and frequency dependence can be eliminated and the instrument can be calibrated relatively easily.

2 APPARATUS

A novel vibrating sample magnetometer was developed at the Department of Materials Science and Engineering of BUTE. This instrument strongly differs from the Foner type magnetometers, because the specimen vibrates along the magnetising field. Among others this parallel motion arrangement has several advantages like in-

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creased sensitivity, simpler detector coil arrangement and easier specimen positioning.

The vibrating system contains an electromagnetic vibrator unit which moves vertically the end of a rectangular vibration rod (Fig.1).

Fig. 1. The electromagnet and the vibrating unit of the PMVSM system

The vibration rod is fixed with a special clamp in the middle and the specimen holder is placed on its free end. The rectangular rod of dimensions 460 x 20 x 6 mm is made of a heat treated AlMgSi alloy which has excellent elastic properties. As a consequence of this construction the sample vibration is parallel to the field. The applied driving signal was sinusoidal with 75 Hz frequency. The laboratory electromagnet of the VSM is driven quasi-statically around the sample's hysteresis loop. A complete loop can be traced in an adjustable period between 2 and 60 minutes.

A PC controlled electronic bipolar voltage regulated current power source controls the DC current of the magnetising vertical electromagnet providing field up to 10^6 A/m. A Hall sensor is built in the lower pole piece to measure the external magnetising field.

The vibrating sample magnetometer is completely PC controlled which offers sophisticated functions including sample demagnetisation, system calibration and graphical user interface. The measured data can be saved as a tab delimited file (txt). The detector coil pair is situated in the centre of the pole pieces. The reference magnet is built in the vibrating rod outside and far from the electromagnet poles. The signals of the detector and reference coils are preamplified with a two channel low noise amplifier and using a 16 bit multi-channel data acquisition card and the special software processed. Taking into account the mass (or volume) of the sample and demagnetising corrections, $M(H)$ or $B(H)$ hysteresis loops can be traced and all the main magnetic parameters can be determined.

Uncommonly, this instrument does not contain hardware lock-in amplifier. The data acquisition software contains a module, which selects the single tone harmonic signal with the highest amplitude in the frequency range of 75 Hz ± 0.1 Hz. This technique was found completely sufficient to remove the noises from signal of the detector coil pair.

Our VSM is designed mainly for practical engineering measurements and for studying induced magnetic anisotropy of materials. The measured specimens are relatively large (typically some hundreds of mg) and made of ferro- and ferrimagnetic materials. The measurements can be completed only at room temperature. Therefore, stable and easy to use equipment with sufficient sensitivity was constructed. This instrument allows us to measure magnetic properties of structural steel specimens used in engineering constructions like steels used in power plants and machine parts. Advantages of the applied construction are the following. Because the sample oscillates parallel with the direction of the magnetic field the induced voltage in the detector coil pair is relatively high which allow us to use a relatively simple detector coil arrangement and it improves the sensitivity and the signal to noise ratio. On the other hand, the parallel sample movement results that there is practically no importance of the homogeneity of the magnetic field between the poles. The replacement and the positioning of the specimen are easy and fast. The reproducibility of the system is good. Sample size can be changed in a relatively wide range. Relative position of the sample to the external magnetic field can be changed. Therefore, the magnetization curves can be studied in different directions consequently the magnetic anisotropy can be studied.

3 SYSTEM PERFORMANCE

The system was calibrated by using a nickel sphere (221 mg) as a standard. Specific saturation magnetisation of nickel is reported to be 54.9 emu/g [3]. The internal magnetic field is related to the applied field via, $H_{internal} = H_{applied} - D \cdot M$ (where: D is the demagnetisation factor of the specimen).

Measurements were done in "step mode" where the magnetic field was changed in discrete steps in every 2 seconds. There was 1950 ms delay after each step and of before the measured data were collected to prevent the transient effects of the magnetic circle. The applied sampling frequency for measuring the signal of the detector coil pair was 100 kHz and the number of measured points was 40 k. The driving frequency of vibration was 75 Hz which made the sample rod vibrated in its first fundamental mode.

The performance of the system was rigorously tested with several series of experiments. The obtained most important system specifications are the following. The accuracy of M and H measurement is better than ±1%. The repeatability of measurement is ±0.5% and magnetic moments (MV) as small as 8.5×10^{-4} emu have been detected. This sensitivity is not outstanding but it was found absolutely sufficient for studying the magnetic properties of structural steels

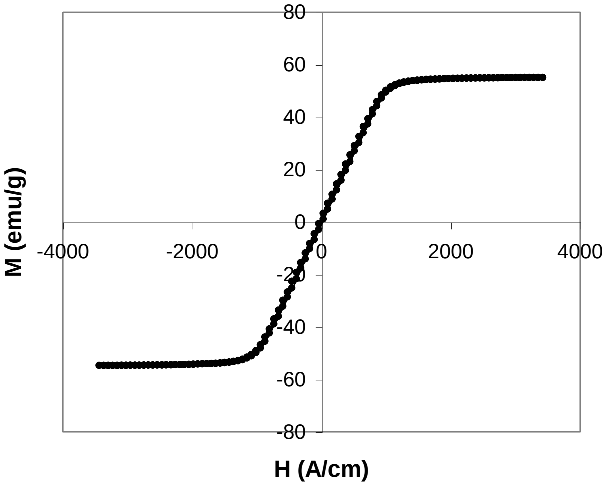

Fig. 2. Measured hysteresis loop of a spherical nickel sphere used for system calibration.

4 EXPERIMENTAL

The investigated 15Mo3 type steel is commonly used as pipeline base material of steam generators in power plants. The investigated steel was hardened (kept at 920°C for 50 minutes and cooled in air) and annealed (kept at 720ºC for 90 minutes and cooled in air). From the original rod material cylindrical creep test specimens were machined with a diameter of 6 and 8 mm for the creep and TSF tests respectively. The specimens have proper heads for fixation in the creep testing instrument.

Tensile creep tests were performed at 550 °C for different times up to 3021 hours. The applied tensile stress was 25 MPa. The sample which was creep tested for 3021 hours was broken at the end of the creep process. The details of the creep tested samples can be seen in Table 1.

Thermal shock fatigue (TSF) tests were also performed. The specimens were clamped stiffly at their both ends in the thermal shock fatigue testing equipment. The TSF samples were heated up by the Joule-heat of an electric current forced through them and when their temperature reached the preset 550 °C value they were quenched by cold water. The temperature was measured by a thermocouple assembled in the middle of the specimens. Heating and cooling cycles last about 30 and 10 second respectively. The total lifetime of the tested steel is defined with the number of cycles at which visible (about 0.1 mm) macroscopic cracks appear on the surface of the specimens. In case of the tested steel the total lifetime was about 4000 cycles. The applied numbers of thermal shock cycles in the present investigation were 0, 100, 300, 530, 1000, 2000 and 4000.

From the middle parts of cylindrical creep samples small cube (edge of 3mm) samples were machined for the PMVSM measurements.

The parallel motion vibrating sample magnetometer was applied for measuring the initial magnetization and saturation hysteresis curves. The samples were completely demagnetised before each measurement to ensure the same initial magnetic state before magnetization process and they were magnetised up to 4500 A/cm external field. The cubic shaped samples were measured in two directions. The initial magnetization M(H) and hysteresis curves corresponding to the longitudinal and transversal directions of the creep specimens were recorded. In this work the longitudinal induced magnetic anisotropy and the coercivity were studied and evaluated.

Tab. 1. Details of the creep tested samples.

Creep time (h)	Remaining life
0	100%
354	88.3%
618	79.5%
821	72.8%
978	67.6%
1127	62.7%
1456	51.8%
1882	37.7%
2225	26.3%
2513	16.8%
2877	4.8%
3021	0%

5 RESULTS

In the initial state the magnetization curves were found nearly the same in longitudinal and in transversal directions as well. Therefore, initially isotropic magnetic behaviour was detected.

Strong dependence of the initial magnetization and hysteresis curves were detected in longitudinal direction.

The width of hysteresis loops decreased continuously due to creep process (Fig.3). Therefore, continuous magnetic softening during the creep process was detected. The decrease coercivity due to creep is considered to reflect the increase in sub-grain size as well as the diffusion of dissolved carbon from a matrix to the boundary of cell or sub-grain.

The increase of creep time decreased the slope of the longitudinal M(H) curves. Therefore, the detected longitudinal initial permeability was found to be decreased by the increase of creep time. As it can be seen in Fig.3 significant and monotonous increase of induced magnetic anisotropy was detected due to the microstructural changes caused by creep process. The creep induced magnetic anisotropy is supposed to be caused by the elongation of grains and the change of stress state. Therefore, appearance of shape and stress anisotropy is supposed.

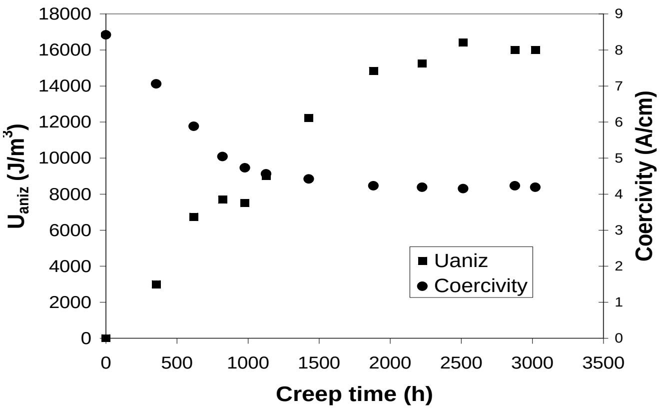

Fig. 3. Coercivity and induced magnetic anisotropy due to creep process

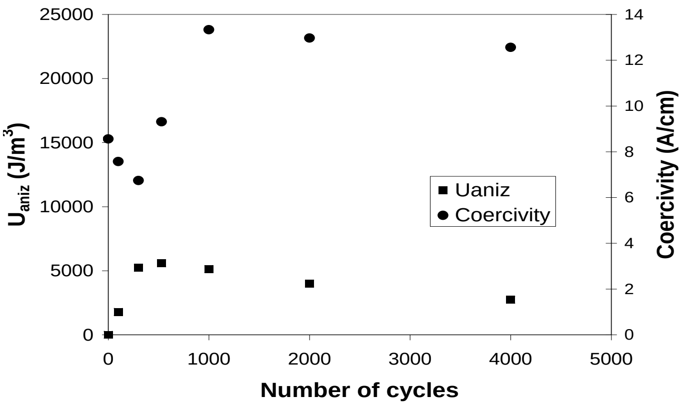

Fig. 4. Coercivity and induced magnetic anisotropy due to thermal shock fatigue process

The TFS process caused cyclic softening in the first 300 cycles as it can be seen in Fig.4. In the range of 300-1000 cycles the increase of coercivity is caused by the increasing dislocation density and the decrease of sub-grain size. This is because, dislocation chains which can act as sub-grain walls can effectively delay the movement of domain walls. Above 1200 cycles the appearing voids and microcracks lead to stress relaxation which causes a slight decrease of coercivity in that range. It is remarkable that the induced magnetic anisotropy only a slightly influenced by the microstructural changes caused by the TSF process.

5 CONCLUSIONS

A new type of vibrating sample magnetometer (PMVSM) was designed and developed at the Department of Materials Science and Engineering. This instrument differs from the Foner type magnetometers because the specimen vibrates along the magnetising field. This parallel motion arrangement has several advantages like increased sensitivity, simpler detector coil arrangement and easier specimen positioning. This stable and easy to use equipment with sufficient sensitivity is designed for practical engineering measurements and for educational purposes. Its excellent performance was demonstrated.

Changes of magnetization curves of 15Mo3 ferritic steel subjected to a tensile creep and thermal shock fatigue processes were studied. Strong dependence of the initial magnetization and hysteresis curves were detected in longitudinal direction.

The creep process caused continuous magnetic softening and significant increase of magnetic anisotropy. In contrast to the creep the thermal shock fatigue process caused magnetic hardening and it has only a slight effect on the magnetic anisotropy.

These findings can be especially important in material testing and useful for developing novel nondestructive magnetic testing techniques.

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REFERENCES

[1] GINSZTLER, J. — DÉVÉNYI, L.: European J. of Mechanical Engineering, Vol. 36 (1991), pp. 251-256.

[2] FONER, S.: Rev. Sci. Instrum, Vol. 30 (1959), pp. 548-557.

[3] GRAHAM Jr., C.D.: J. Appl. Phys, Vol. 53 (1982) pp. 2032-2034.

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