Ageing behavior study of 5Cr–0.5Mo steel by magnetic Barkhausen emissions and magnetic hysteresis loop techniques


a National Metallurgical Laboratory, Jamshedpur, India
b School of Materials Science and Engineering, Bengal Engineering and Science University, Howrah 711 103, India

Received 23 June 2006; received in revised form 1 August 2006; accepted 31 August 2006
Available online 30 October 2006

Abstract

Magnetic hysteresis loop and Barkhausen emissions were recorded and analysed for 5Cr–0.5Mo steel after ageing at 600 °C for various lengths of time. At the initial stage of ageing the interstitial carbon diffuses towards the grain boundary making the matrix magnetically softer. During this stage, extending up to 200 h of ageing, magnetic softness was found to be increasing. This was associated with a decrease in coercivity and increase in Barkhausen voltage. Beyond 200 h of ageing the precipitation of alloy carbides attain subsequent growth, making the material magnetically harder. The evolution of carbides was studied using SEM-EDAX analysis. It was observed that most of the carbides transformed into M23C6 type after ageing for 400 h.

Keywords: Magnetic Barkhausen emissions; Magnetic hysteresis loop; Ageing; Microstructure; Hardness

1. Introduction

Subjected Cr–Mo steels are widely used in power plant and petrochemical industries as tubing materials to high temperature (350–600 °C) and pressure (15–30 MPa). These are expected to sustain at least for 250,000 h [1]. Therefore, in recent years there have been significant efforts in industries to determine the remaining life of the components approaching their design lives. This requires an in-depth knowledge of the influence of long term high temperature exposure on the microstructure and on the mechanical properties [2]. Extensive microstructural analysis has been carried out to understand the metallurgical changes that take place during ageing [3–6]. Different microstructures have also been simulated to characterize heat-affected zone (HAZ) and also the weldments [7,8]. Major emphasis on these studies has been concentrated on the evaluation of microstructure and its co-relation with mechanical properties of the materials [9–11]. Only a limited number of investigations have been carried out to understand the influence of microstructure on the physical properties of the high temperature Cr–Mo steel materials. Such studies are necessary to identify suitable parameters useful for damage assessment of the in-service component by non-destructive way.

Cr–Mo steel is ferromagnetic in nature. Its magnetic properties change with microstructural changes that take place during in-service operation. The magnetic properties depend on the grain size, chemical composition, the nature and distribution of precipitation. The fact that a few of their characteristics would change during in-service operation makes this technique a potential tool for the damage assessment of the engineering components. However, many of the metallurgical changes taking place during in-service may have reverse effect on the magnetic properties. For example, grain growth that usually takes place during high temperature operation makes the material magnetically softer whereas of non-magnetic phases precipitation makes the materials magnetically harder. This would mask the effect of damage on magnetic properties we want to measure. Thus, there is a need for detailed study on effect
of stress and temperature on magnetic properties of the engineering components. The present investigation reports the effect of thermal exposure on the magnetic properties of 5Cr–0.5Mo steel. Determination of magnetic hysteresis loop and analysis of magnetic Barkhausen emissions form the major part of this study.

2. Material

The material for the present study is 5Cr–0.5Mo steel obtained from petrochemical industry in the form of a 10 mm thick tube with 100 mm diameter. Flat specimen of 100 mm length, 25 mm width 4 mm thick were cut from the tube components for the present study. The chemical composition of the as-received tube is shown in Table 1. Fig. 1 shows the microstructure of the as-received sample which is bainitic in nature with ferrite in dark areas and carbide in white areas. The microstructure revealed that the as-received material is in its virgin state. It has not been put in service.

3. Experimental

The samples were aged at 600 °C for various durations. Microstructures of the aged sample were examined under a JEOL JSM 840A scanning electron microscope (SEM) with an attachment for energy dispersive X-ray micro-analysis (EDX). The etch medium used for the micro-structural study was picral having 100 ml ethanol, 2 gm ferric chloride, 4 gm picric acid and about 10–15 ml HCl. Vickers hardness was measured in a computerized hardness testing instrument with 3 kg load. Magnetic hysteresis loop and Barkhausen emissions were measured using surface magnetizing probe. Magnetic hysteresis loop was plotted at a quasi-dc (50 mHz) magnetizing field whereas the Barkhausen emissions were measured at 40 Hz using 30–300 kHz band pass filter.

4. Results and discussion

4.1. Microstructure and hardness

Fig. 2 shows the Vickers hardness of the material aged for different hours. The hardness of the material decreased from 142 VHN with ageing. It reaches minimum of 126 VHN at 200 h of ageing and then started increasing. Initial microstructure indicated that grains were of irregular shape and carbides were observed both at the grain boundaries and at the grain interiors. After 1 h of ageing there was no significant change in microstructure. However, with progressive ageing although there was no significant grain growth taking place, noticeable changes were observed in carbide composition, size and morphology. The change in the nature and distribution of carbides with ageing, and the representative microstructure of the samples aged for 1, 200 and 400 h are shown in Fig. 3–5, respectively. Initially, the carbides were mostly found at the grain interior (Fig. 3a). SEM micrograph at higher magnification showed (Fig. 3b) that the carbides were fibrous type. These carbides were mainly M2C, M3C, M7C6.
type where M stands for Fe or Cr or Mo or a combination of them. EDAX analysis of a typical carbide composition is shown in Fig. 3c which is possibly M₇C₆ type of carbide.

On ageing there will be significant partitioning of carbon. The diffusion co-efficient of interstitial carbon is several order of magnitude faster than the diffusion co-efficient of substitution elements like Cr, Mo. This large difference in diffusion coefficients allowed carbon to diffuse faster towards grain boundary. The substitution elements need more time to redistribute during transformation between phases even though there is a significant partitioning of carbon. Thus, there was a decrease in carbon content within the matrix with ageing. Fig. 4a shows the microstructure with 200 h of ageing. This shows an increase in amount of grain boundary carbides. The morphology of the carbides at the grain interior also changed as shown in Fig. 4b. The fibrous type of carbides within the grain slowly got spheroids. The carbide composition also changed as shown in the EDAX analysis (Fig. 4c). The microstructure of the sample after 400 h of ageing is shown in Fig. 5a. In this case the grain boundary carbide increased and carbides at the grain interior became spheroids (Fig. 5b). The composition of the carbides also changed and became M₂₃C₆ type as suggested from EDAX analysis (Fig. 5c).

4.2. Magnetic properties

Magnetic hysteresis and Barkhausen parameters were evaluated for different aged materials. The results were normalized with respect to sample aged at 1 h so that any effect of stress that was generated during sample preparation on magnetic properties would be eliminated. Fig. 6 shows the variation of normalized coercivity with respect to ageing time. Coercivity started decreasing after 10 h of ageing and became a minimum at 200 h of ageing. Thereafter it kept on increasing with ageing. This indicated that magnetic softness increased with the ageing up to 200 h and subsequently its magnetic hardness increased. This was also corroborated with the magnetic Barkhausen emissions study. Fig. 7 shows the magnetic Barkhausen emissions waveform for a few selected samples after 1, 200 and 400 h of ageing. The rms voltage of the Barkhausen emissions shown in Fig. 8 increased with the ageing period up to 200 h, indicating magnetic softness. Thereafter, rms voltage decreased with ageing. There was no significant change in
grain size due to ageing. Therefore, its effect on the magnetic properties was ignored. However, only at the initial period of ageing, the dissolved carbon diffused towards grain boundary, leaving the matrix less strained and thereby increased magnetic softness by reducing magneto elastic anisotropy. It is well known that the soft magnetic nature of steels gets enhanced as carbon content decreases [12]. Thus, the depletion of interstitial carbon from the matrix due to its migrating towards grain boundary, made the steel softer at the initial stage of ageing. This reduction of carbon also allowed the domain wall to move further resulting in reduction of low amplitude Barkhausen pulses and an increase of high amplitude Barkhausen pulses. This behaviour of pulse height distribution has been reflected in Fig. 9, where a comparison of pulse height distribution has been made for different hours of ageing. As the duration of ageing was increased, the fibrous type of carbides started breaking and became spheroids. Thus, a situation existed when two effects appeared simultaneously. The enhancement of soft magnetic properties is due to reduction of carbon from the matrix, and a competing effect of deterioration in soft magnetic properties is due to breaking of fibrous type carbides which increased the pinning density for the domain wall movement. Till 200 h of ageing, soft magnetic properties continue to improve which was observed both from coercivity and rms voltage of Barkhausen emissions measurement. It was observed from the pulse height distribution that for 200 h aged sample the number of high amplitude pulses as well as the low amplitude pulses increased from 6 h aged sample. This increase in numbers of both high and low amplitude pulses as compared to 6 h of aged sample indicating that in case of 200 h aged sample both the effects of magnetic softness due to reduction of interstitial carbon in the matrix as well as magnetic hardness due to pinning of domain wall by the carbides at the grain interior took place. As a result of ageing the disintegrated fibrous type of carbides, getting spheroids with associated change in the size and composition. Further ageing led to transformation of M23C6 types of carbides. Newer carbides were also formed with ageing. These carbides not only exert pinning forces but also produce large demagnetizing field which retarded the domain wall motion making the material magnetically harder. Therefore, the coercivity of the materials increased and rms voltage of the Barkhausen emissions decreased.

![Fig. 4. SEM micrograph of 600 °C/200 h aged 5Cr–0.5Mo steel sample at (a) low magnification showing the grain boundaries, (b) high magnification showing the carbides within the grain interior, (c) SEM–EDX analysis of the carbides within the grain interior.](image)
when the ageing time was greater than 200 h. The pulse height distribution also showed increase in low amplitude pulses after 400 h of ageing. Thus the magnetic hardness for 5Cr–0.5Mo steel at higher ageing time was due to the formation of M23C6 types of carbides at the grain interiors.

5. Conclusions

Magnetic hysteresis loop and Barkhausen emissions were evaluated for 5Cr–0.5Mo steel aged at 600 °C for various hours and the results were correlated with the microstructure of the samples. Magnetic softness for materials below 200 h of ageing was primarily due to the depletion of carbon from the matrix which migrated towards the grain boundary. As soon as the carbides within the grain interior start growing magnetic hardness is observed. Microstructural evidence showed that with ageing, the carbides within the grain interior slowly transformed to M23C6 type of carbides. This transformation and growth of carbides made
the materials magnetically hard. The increase of low amplitude pulse in pulse height distribution of Barkhausen emissions for 400 h aged sample indicated the hindrance of domain wall by the carbides resulting in the magnetic hardness. The present study showed a good correlation between variation in magnetic properties and corresponding microstructural change. This investigation further strengthens the scope of magnetic measurement techniques for evaluation of microstructural change during ageing of in-service 5Cr–0.5Mo steel components.

Acknowledgement

The authors are grateful to the director NML for his permission to carrying out the work and publishing the paper.

References