Magnetic evaluation of creep in modified 9Cr–1Mo steel


National Metallurgical Laboratory, Jamshedpur-831007, India

Received 16 May 2007; revised 4 July 2007; accepted 5 July 2007
Available online 2 August 2007

Abstract

Magnetic Barkhausen emissions (MBE) and magnetic hysteresis loop techniques have been used to correlate the magnetic properties with creep behaviour in modified 9Cr–1Mo steel. Formation of massive carbides like M₂₃C₆, Laves phase (Fe₂Mo) and Z-phase are the cause of creep failure in the present material. The root mean square voltage of MBE signal increased from the secondary stage of creep. This was attributed to the growth of carbides. Microcracks generated by massive and brittle precipitates induced demagnetizing fields that restricted domain wall movement, resulting in continuous reduction of remanence during creep.

© 2007 Acta Materialia Inc. Published by Elsevier Ltd. All rights reserved.

Keywords: Creep; Barkhausen emissions; Coercivity; Remanence; Microstructure

Modified 9Cr–1Mo steel where Nb and V are added for better high temperature creep resistance is a relatively new structural alloy used in advanced fast breeder reactors, power generation and petrochemical industries within the temperature range of 773–873 K [1,2]. In this temperature range, under an extended period of service, the carbide composition and morphology change, and new phases are formed. Initially, M₂C- and Mo₂C-type carbides that present in the as-received materials dissolve and, with the increase in the service period, other carbides are formed, like M₇C₃, which ultimately transforms to M₂₃C₆ type, where M stands for Fe or Cr or a combination of them. Phases like VN and Nb(C,N) are also formed in the intermediate service period, and these provide a strengthening effect to the materials [3,4]. Although the microstructure and mechanical behaviour of creep-damaged samples have been analyzed by various groups, only a few studies have been made on the influence of creep process on the physical properties, like internal friction and the elastic modulus [5,6]. The aim of the present study is to find the influence of creep on the magnetic properties of modified 9Cr–1Mo steel so that the technique can be useful for damage assessment of in-service components in a non-invasive manner for estimating remaining life.

An experiment was conducted on a standard flat specimen of 25 mm gauge length cut from a 9Cr–1Mo virgin tube having the nominal composition shown in Table 1. A creep test was carried out at 873 K with a stress value of 125 MPa. The test was interrupted at specified intervals for taking magnetic measurements. Magnetic hysteresis loop (MHL) measurements and magnetic Barkhausen emissions (MBE) were taken at the gauge length region of the sample using a surface probe under a magnetizing field of 80 kA m⁻¹ for MHL and 4 kA m⁻¹ for the MBE measurement [7]. The magnetizing frequency for the MHL study was 50 mHz, whereas 40 Hz magnetizing frequency was used for the MBE measurement. The band pass filter used for the MBE measurement was 30–300 kHz as most of the signals were within this frequency range.

Figure 1 shows a transmission electron micrograph of the modified 9Cr–1Mo steel in its virgin state, which is tempered martensitic in nature. The carbides were found at the grain and lath boundaries as well as inside the lath structure. Indexing the selected area diffraction (SAD) pattern shows that most of the carbides are M₂C- and M₇C₃ types (where M stands for a Cr-/Fe-like metal or a combination of them).

Figure 2 shows a scanning electron micrograph of the fracture surface of the failed material. The presence of a large number of dimples in the fractured surface suggests that the material became ductile before fracture and energy-dispersive X-ray analysis at the dimples indicated the presence of a Cr-rich carbide. Those carbides are pulled out when the strength of the material falls drastically. A transmission electron micrograph of the creep-ruptured material close to the fractured end is shown in Figure 3a. Due to creep, the martensitic laths become wider and the carbides became coarser. These
carbides were predominantly of the M23C6 type, and were distributed at the grain and lath boundaries. The morphology of a typical Cr23C6 type of carbide is shown in Figure 3b. Figure 3c shows the overlapping SAD pattern of the predominant carbide with a cubic structure, having indices (222), (440), (222) and a zone axis Z = [110]. The overlapped SAD patterns are from the martensite (BCT structure) matrix, having indices (101), (110), (011), with a zone axis Z = [011] and from carbide Mo2C (orthorhombic structure), having indices (121), (114), (033), with a zone axis Z = [110]. Besides Cr-rich carbides, the Mo-rich phase known as Laves phase (Fe2Mo) was also observed in the creep-damaged sample and is shown in Figure 3d. This phase has a hexagonal structure, with indices (112), (220), and (112) with a zone axis Z = [110]. Besides Cr23C6 and Fe2Mo, a complex carbonitride phase, Cr(VNb)CN, was also observed in the creep-damaged sample which was identified as Z-phase [8,9].

Figure 4 shows the average value of the root mean square (rms) voltage of the Barkhausen emissions against the percentage of expended creep life. The results in the figure have been plotted after normalizing the rms voltage with respect to the rms value at 50 h of creep testing, which was the first available data point. The
creep strain is also plotted with expended creep life in the same figure to understand the mechanical behaviour of the sample undergoing creep testing. According to the variation in creep strain rate, the creep life is divided into three regions: (i) the primary creep region; (ii) the secondary creep region; and (iii) the tertiary creep region. In the present material, 25% of the expended life was spent in the primary creep region, the following 45% in the secondary creep region and the remaining 30% in the tertiary creep region, as shown in Figure 4.

The study revealed that the rms voltage of Barkhausen emissions signal decreased in the primary creep region. It started increasing in the secondary creep region, and subsequently the rate of increase of rms voltage decreased during tertiary creep. The pulse height distributions of the Barkhausen signal obtained from different stages of creep are compared in Figure 5. More low-amplitude Barkhausen pulses were found at the beginning of the secondary region than in the primary region. This is illustrated in Figure 5a, where the distribution for 40% expended creep life is compared with the 4% expended creep life of the primary region. As the time of exposure increases in the secondary region, the high-amplitude Barkhausen emissions signals started increasing slowly. Figure 5b shows the slow variation in pulse height distribution when the Barkhausen signal for 60% expended creep life was compared with those after 40% expended creep life. However, in the tertiary region a rapid increase in high-amplitude Barkhausen emissions was observed, as shown in Figure 5c, where signals distributed after 85% expended creep life is compared with those after 60% expended creep life.

The hysteresis loop parameters, i.e. coercivity and remanence, are plotted in Figure 6 after normalizing with the corresponding values of 50 h of creep testing. Coercivity (Hc) and remanence (Mr) were both found to increase during the primary creep for the studied modified 9Cr–1Mo steel used in this study. In the secondary creep region both Hc and Mr started decreasing. However, in the tertiary creep region Hc increased very rapidly whereas a continuous decrease in Mr was observed.

At high temperature, the dissolved carbon within the material diffuses towards the grain boundary, leaving the matrix less strained and depleted in interstitial carbon content, which influences the magnetic properties of the steel [10,11]. The kinetics of this process becomes faster when the material is under stress, i.e. under creep. During the migration towards the grain boundaries in the primary stage of creep, carbon interacts with the existing carbides like M_2C and M_3C_2, where M stands for Fe, Cr or their combination. This is likely to change the carbide composition and morphology. The coarsening of carbides at high temperature gives a small number of widely spaced pinning centres. At high temperature carbon also interacts with the other alloying elements and new carbides, like VC, NbC and Mo_2C, are formed. Nitrides, like VN and NbN, are also formed at this high temperature [12,13]. The precipitation of fine carbides and nitrides, which increase the
creep strength, also act as pinning centres, restricting the magnetic domain wall movement. Such restrictions increase the number of low-amplitude pulses in Barkhausen emissions, as shown in Figure 5a, when the 4% expended creep life sample was compared with that of 40% expended creep life. Precipitation of new carbides and nitrides also increases the coercivity and decreases the rms voltage of the Barkhausen signal. In other words, magnetic hardening was observed in the primary creep region. This suggests that in the primary creep region formation of fine carbides and nitrides affect the magnetic properties predominantly compared with the effect of the coarsening of carbides. As the creep moves to the secondary creep region, the size of the carbides becomes larger at the expense of the smaller ones, reducing the number of pinning centres and increasing the spacing between them. Hence pinning density decreases with progressive accumulation of creep strain. This is also reflected in the decrease in coercivity and increase in rms voltage of the MBE signal. The materials with longer creep exposure (say, 60%) generate more high-amplitude pulses in comparison with those with lower creep exposure (say, 40%) as the domains could move to longer distances without being pinned by carbides (Fig. 5b). As creep progresses further, massive carbides, like M\textsubscript{23}C\textsubscript{6}, are formed (Fig. 5b). This decreases the low-amplitude pulses further and increases the high-amplitude pulses (Fig. 5c). In this creep region, massive brittle phases, like Laves phase (Fe\textsubscript{2}Mo) and Z-phase (a complex carbonitride phase of Cr, V and Nb), are also formed, as shown in Figure 3c [1,9]. These brittle phases reduce the creep strength and lead to microcracks at the grain boundaries, causing material failure [14]. In the primary and the secondary creep regions, the behaviours of coercivity and remanence are similar (Fig. 6). However, in the tertiary region a decrease in remanence and increase in coercivity were observed. This was primarily because of the demagnetization field resulting from the non-magnetic massive carbides and other phases generated in the materials during the extended period of creep exposure. Due to the formation of microcracks, the domain wall movements become restricted, as indicated in the slow change in rms voltage and increase in coercivity.

To conclude: the magnetic Barkhausen emissions and magnetic hysteresis loop techniques were used to correlate magnetic properties with the creep life fraction, with the aim of determining whether the techniques could be used for the non-destructive evaluation of creep damage. A good correlation between microstructures and magnetic parameters (both Barkhausen and hysteresis) was observed during the different stages of creep. The formation of massive M\textsubscript{23}C\textsubscript{6} carbides, Laves phase and Z-phase is found to be the cause of creep failure in modified 9Cr–1Mo steel. The present study also indicates that rms voltage of Barkhausen emissions signal can be used as a suitable non-destructive magnetic parameter for assessing creep damage in this steel.