

Regular article

Oxidation kinetics in pearlite: The defining role of interface crystallography

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ABSTRACT

Five different pearlitic steel wires were produced through controlled thermomechanical processing. These wires had significant (~2 times) differences in the pearlite alignment and also exhibited nearly one order of magnitude difference in the oxidation kinetics. Though the nature of the oxide films was not remarkably different, the stresses in the majority hematite phase scaled with the experimentally measured oxidation rates. Careful correlation between substrate microstructure and oxide stresses established that the stresses were highest at the ferrite–cementite interface. And the interface crystallography, in case of the coarse pearlite, determined such stresses and the oxidation kinetics.

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Oxidation, reaction between metal and oxygen [1,2], has tremendous implications for the processing and performance of iron and steel. These may include oxidation losses during thermomechanical processing (TMP) to pickling or the technological aspects of oxide removal [1–3]. Naturally, efforts are also made [1,4–6] to tailor alloy composition for relative oxidation resistance.

Iron oxides exist as different phases: Wüstite (FeO), followed by Magnetite (Fe₃O₄) and Hematite (Fe₂O₃) [1,2,7]. The presence of these phases depends on the oxidation temperature [1,2,7], and also on the environment [8,9]. It has been argued [1,2,7] that the growth of individual oxide layers takes place by ionic transport. Diffusion and availability, of the respective ions and their electronic structures, are thus of large importance [1,2,7]. There is no surprise that the atmosphere, or the oxygen partial pressure at the solid–vapor interface, has an important role to play [8,9]. Similarly, the chemistry of the alloy may determine availability and nature of the metallic ions, and hence affect oxidation [4,10–13].

Past researchers have tried to relate oxidation behavior with the morphology [7,14] and microstructure [7,10,14] of the iron oxide. However, relatively “limited” attempts were made in appreciating the oxide micro-texture [2,7] and the state of residual stress [2,15]. The later, for example, may offer significant insights. The stress in the oxide decides

its adherence and influences the diffusion [2,15]. Though its importance has often been acknowledged [7], there is a clear lack of comprehensive studies relating stresses in the iron oxide with the substrate microstructure and linking them together to the overall oxidation behavior. And this was the motivation behind the present study.

This study involved fully pearlitic near eutectoid steel: a material with a wide range of technological applications [16]. The mechanical performance, tensile as well as torsional, of this class of “nanocomposite” depends on the interlamellar spacing and the pearlite alignment [16–18]. A recent study [19] had shown that controlled TMP, imposing different cooling rates and/or deformation during austenite-to-pearlite phase transformation, may significantly alter the pearlite alignment. And this provided the starting material for this manuscript. As shown in Fig. 1a, between the 5 selected wires the pearlite area fraction versus morphological angle (with the wire axis) were remarkably different. The relative pearlite alignment, percentage of pearlite colonies within 20° of the wire axis, nearly doubled from 31% to 66% (see Fig. 1b). This was accompanied by a relatively minor, albeit consistent, change in pearlite interlamellar spacing, from ~220 to ~200 nm [19], and more noticeable changes in ferrite crystallographic texture (Fig. 1b). In a word, pearlite microstructure (especially the morphology) was significantly different (see Fig. 1a–b) in the longitudinal sections of the 5 different wires. These wires were then used for subsequent oxidation and oxide characterization.

Fig. 1c shows the relative weight gain data from a thermogravimetric analyzer (TGA), TA50™. TGA measurements were conducted in an atmosphere of 1% oxygen and ~99% nitrogen and

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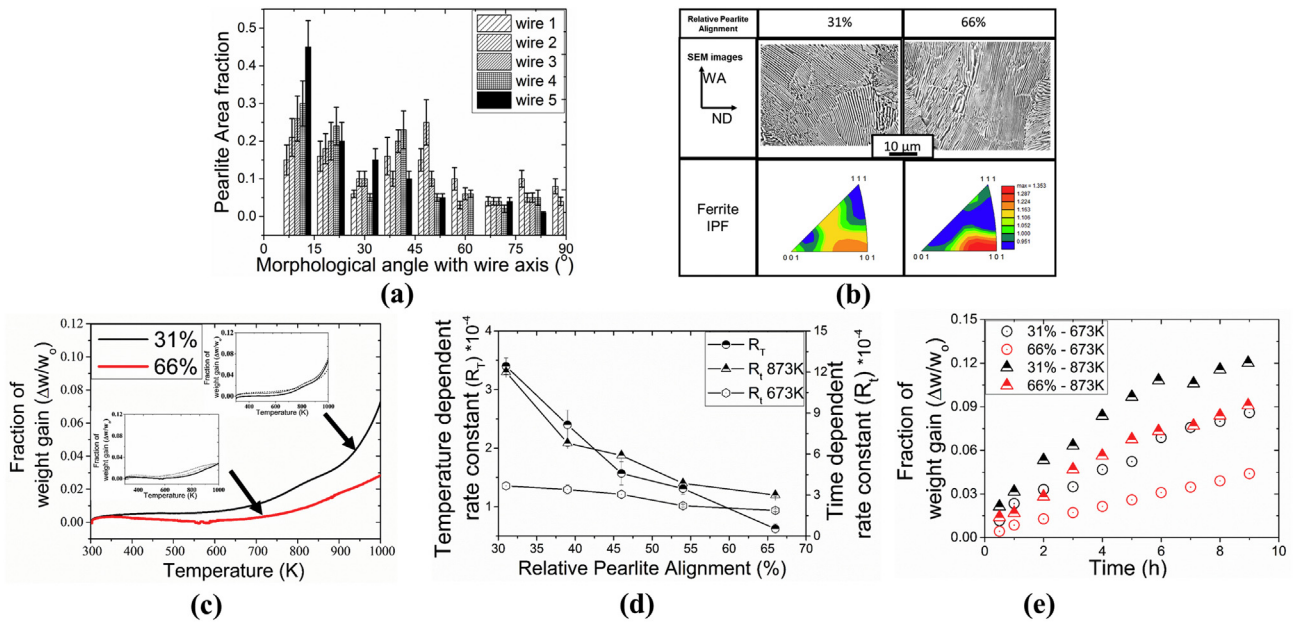


Fig. 1. (a) Pearlite area fraction versus morphological angle for 5 different pearlite wires (wire 1 to 5). Relative pearlite alignment was estimated from the area fraction of pearlite colonies within 20° of the wire axis. (b) SEM (scanning electron microscope) micrographs (WA and ND representing wire axis and normal direction, respectively) and ferrite crystallographic textures (in IPF or inverse pole figure notation) for pearlite wires with 31% and 66% relative alignment. (c) Fraction of weight gain ($\frac{\Delta w}{w_0}$) versus temperature for differently aligned pearlite wires. Inset shows 3 measurements on wires of extreme alignment (31 and 66%): to confirm reproducibility. (d) Temperature (R_T) and time (R_t ; for 673 K and 873 K holding) dependent rate constants versus relative pearlite alignment. (e) Fraction of weight gain versus time for long term exposure (up to 9 h duration at 673 K and 873 K) of 31% and 66% aligned wires.

10 K/min heating rate. As shown in the figure, $\frac{\Delta w}{W_0}$ (Δw and w_0 are the weight gain and the initial specimen weight, respectively) was lower for the wires with higher alignment. It also needs to be noted that

such difference in oxidation kinetics, with pearlite alignment, was consistent and reproducible, see inset of Fig. 1c. The TGA data was further exploited to calculate temperature dependent rate constant

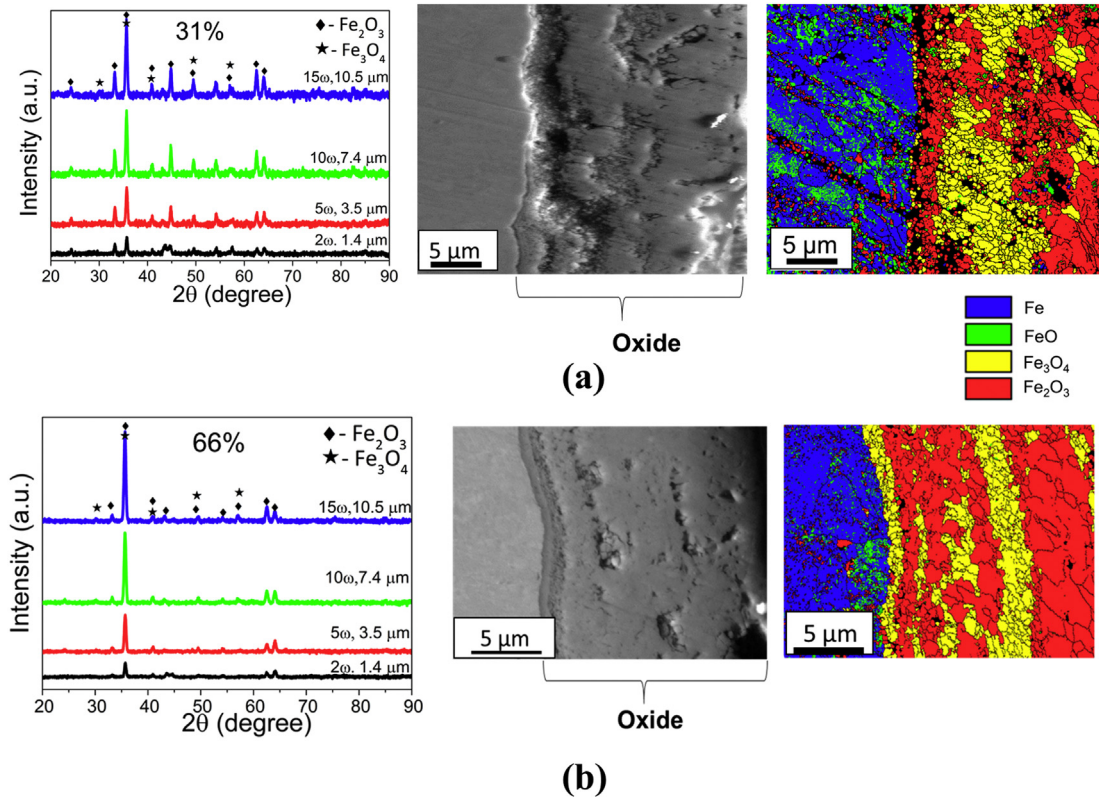


Fig. 2. Oxide (post 873 K-9 h oxidation) characterization with grazing incident X-ray diffraction (GIXRD) and electron backscattered diffraction (EBSD). These are shown for the respective relative pearlite alignment of (a) 31% and (b) 66%. In the GIXRD, grazing angle (ω) was changed to achieve different depth of penetration and the corresponding oxide peaks were identified. Cross-sectional SEM plus EBSD also identified the respective oxide phases. This is shown in color code, with black indicating indexing below 95% accuracy. (For interpretation of the references to color in this figure, the reader is referred to the web version of this article.)

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