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Effect of thermal aging on grain structural characteristic and Ductile-to-Brittle transition temperature of CLAM steel at 550 °C

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HIGHLIGHTS

- The grain boundary length per unit area decreased with the increasing aging time.
- The fraction of LABs increased obviously after thermal aging.
- Prior austenitic grain refinement is more important to improve low temperature toughness.

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ABSTRACT

In this work, electron backscatter diffraction (EBSD) was used to investigate the grain structure evolution of China low activation martensitic (CLAM) steel samples which were aged at 550 °C for 0 h, 2000 h, 4000 h and 10,000 h. The results showed that the prior austenitic grain size increased with the aging time, which led to the decrease of grain boundary length. The fraction of misorientation angle in a range from about 4 to 10° increased obviously after thermal aging for 10,000 h, and it indicated that the fine subgrains formed in the CLAM steel during the long-term thermal exposure. Furthermore, Charpy impact experiments were carried out to analyze the toughness of the CLAM steel before and after aging, particularly the Ductile-to-Brittle Transition Temperature (DBTT). Though amounts of fine subgrians formed in matrix, a substantial increase in DBTT (~40.1 °C) had been noticed after aging for 10,000 h. The results showed that the high angle boundaries such as prior austenitic grain boundaries are more effective in retarding the propagation of cleavage crack than subgrain boundaries.

1. Introduction

China Low Activation Martensitic (CLAM) steel, one of the Reduced Activation Ferritic/Martensitic steels (RAFMs), has been developed by Institute of Nuclear Energy Safety Technology, Chinese Academy of Sciences (INEST, CAS) [1–6]. A series of R&D activities on CLAM steel have been done to investigate the mechanical properties [7,8], irradiation properties [9], fabrication techniques of Test Blanket Module (TBM) [10,11], etc. Due to outstanding properties, the CLAM steel has been chosen as the primary structural material in the FDS series PbLi blankets for fusion reactors [12–19], breeder blanket of China Fusion Engineering Test Reactor (CFETR) and Chinese TBM for International Thermonuclear Experimental Reactor (ITER).

The temperatures of CLAM steel which is used in breeding blanket are proposed to rang from 480 °C to 550 °C and thermal damage is one of the main factors to affect the operational embrittlement of the steels in nuclear power industry [20–22]. Hence, thermal stability behavior of CLAM steel is important to ensure the operation safety after long-term application in reactors. During the long-term thermal exposure in the operation temperature, microstructural evolution of CLAM steel will occur, resulting in changes of mechanical properties. Our previous investigation on the microstructure evolution of CLAM steels after thermal aging were mainly focused on the coarsening of primarily $M_{23}C_6$, nucleation and subsequent growth up of Laves-phase and the recovery of martensitic laths [24]. However, during the thermal aging, the formation of subgrains and migration of martensitic lath boundaries in CLAM steel would influence the distribution of misorientation angle [23–27]. Moreover, grain structural characteristic, such as the misorientation angle, grain boundary length and grain size would affect the mechanical properties significantly [26,27].

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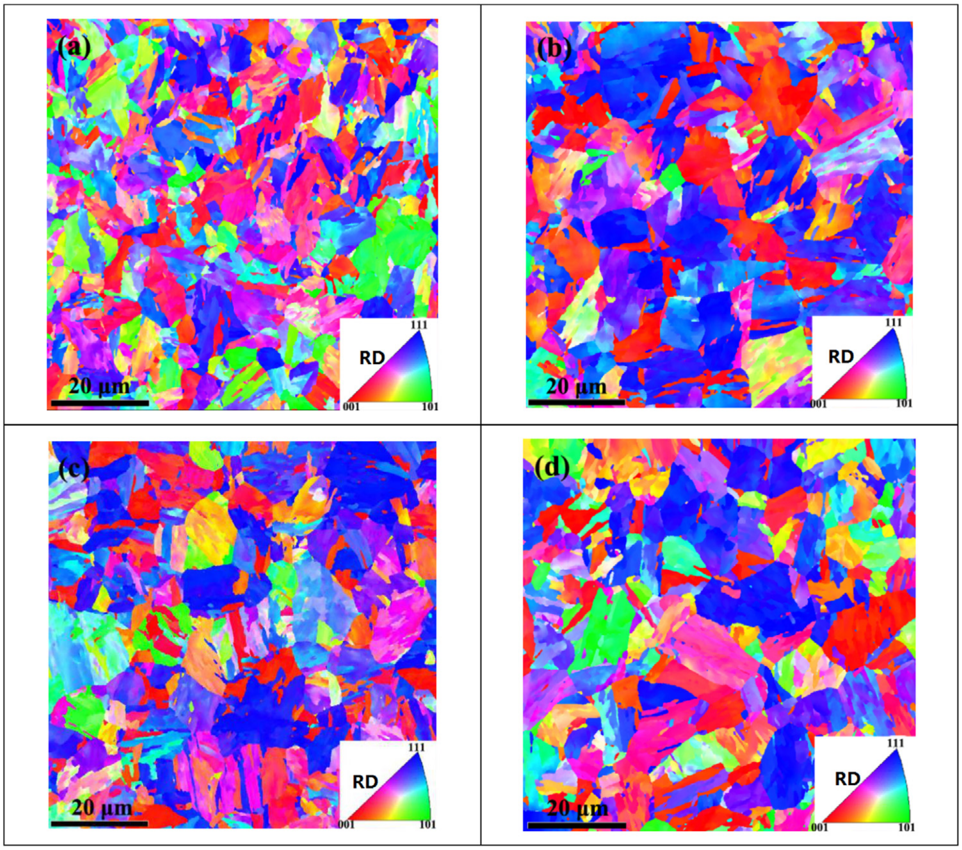

Fig. 1. IPF maps of aged CLAM steel by EBSD: (a) 0 h, (b) 2000 h, (c) 4000 h, (d) 10,000 h.

In general, smaller grain size shows lower DBTT in the ferrite/martensite steels due to higher grain boundary area per unit volume [7]. However, according to other researches on ferritic steel [27], not all grain boundaries are effective in resisting the crack propagation to increase the fracture resistance. This is related to the misorientation angle of grain boundary. The high misorientation angle boundaries are considered to be the effective grain boundary [27]. In order to optimize the microstructure and to improve the low temperature toughness after long-term thermal exposure, it important to investigate the grain structural characteristic evolution and its effects on the impact toughness of CLAM steel during long-term thermal aging. Thus, the present study was focused on the effect of thermal aging on evolution of both grain structural characteristic and impact toughness of CLAM steel.

2. Experimental procedures

The chemical compositions of CLAM steel (HEAT 1005) used in this study was 0.092%C, 8.9%Cr, 0.14% Ta, 0.15% V, 1.5% W, 0.05% Si, 0.49% Mn, 0.005% P, 0.002% S, and Fe in balance (in wt.%) [24]. The material was heat treated with normalizing at 980 °C for 30 min followed by air cooling and then tempering at 760 °C for 90 min followed by air cooling. The aging experiments were carried out at 550 °C with periods for 0 h, 2000 h, 4000 h and 10,000 h under air atmosphere. In order to remove the oxidation effects on the mechanical properties, the machining allowance of each sample was more than 2 mm.

EBSD technology was used to analyze the grain structural characteristic of CLAM samples before and after aging. All the surfaces of samples for EBSD analysis were taken perpendicular to rolling direction (RD). In order to obtain high quality EBSD data, the samples were finally polished with an alkaline colloidal silica solution for 4 h to remove mechanical distortion. The EBSD measurements were carried out with ZEISS ΣIGMA Field Emission Scanning Electron Microscope (FE-SEM) equipped with an Oxford NordlysMax^2 EBSD detector. A selected area of $80 \times 80\ \mu m^2$ combination with a step size of 0.1 μm was used for reasonable statistics of temper martensitic microstructure. Microstructures were also analyzed with transmission electron microscope (TEM; FEI Tecnai G2 F20 S-TWIN).

The DBTT was obtained from Charpy impact tests with testing temperature ranged from −120 °C to room temperature (RT). Not less than two samples were tested for each condition. The testing samples were V-notch with a size of $10 \times 10 \times 55\ mm^3$. All the samples for Charpy impact tests were taken along rolling direction.

3. Results

3.1. Grain structural characteristic

The grain structural characteristics of CLAM samples after thermal aging for different time are shown in Figs. 1 and 2, which were obtained by EBSD with the magnification of 1000×.

Fig. 1 shows the inverse pole figure (IPF) maps for CLAM samples. The standard triangle in each maps shows the crystallographic poles aligned with rolling direction of the samples. Different colors in IPF maps represent different orientations according to the standard triangle. After aging 550 °C up to 10,000 h, the IPF maps revealed that grain structures were changed inconspicuously.

The grain boundaries of the area in Fig. 1 are shown in Fig. 2. Different colors in the grain boundary maps represent different boundary misorientation angle. Red lines in Fig. 2 represent the low-angle boundaries (LABs) of 2–10° misorientation angles, and blue lines represent high-angle boundaries (HABs) of higher than