A comparison of microstructure evolution due to fatigue loading in Eurofer 97 and ODS Eurofer steels

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Abstract

The Eurofer 97 steel and its variant strengthened by fine oxide dispersion (ODS Eurofer) were investigated. Both variants were subjected to the cyclic loading at room temperature. Moreover, the Eurofer 97 steel was exposed to the cyclic loading at 550°C and the ODS Eurofer steel at 650 and 750 °C. Continuous cyclic softening was found in both variants. Nevertheless, addition of 0.3 wt% of Y$_2$O$_3$ significantly reduces softening rate in the ODS Eurofer compared with the Eurofer 97 steel. Microstructure evolution and the role of oxide dispersion were investigated by means of transmission electron microscopy. It was observed the oxide dispersion suppresses grain growth in the ODS Eurofer steel and thus possible operating temperature is higher. The oxide particles remain stable up to highest temperature studied. The microstructural aspects are discussed in relation to the cyclic behavior of both steels.

Keywords:

1. Introduction

Numerous variants of ferritic-martensitic steels have been produced and investigated. F82H steel and Eurofer 97 steel are the most promising variants. Moreover they belong to the group of materials with reduced activation, which is a necessity for future fusion applications or for usage in fission power plants of IV generation. Their mechanical properties have been widely studied. One important drawback is...
continuous cyclic softening which is characteristic for both steels at any testing temperature and also for their older variants P91, P91, Optifer etc. Recently, small amount of fine yttrium oxides were added into microstructure of the Eurofer 97 steel in order to improve strength, remove cyclic softening and stabilize microstructure at elevated temperatures.

This paper deals with low cycle fatigue of the Eurofer 97 steel and its ODS variant, ODS Eurofer steel. Softening curves measured at room temperature (RT) and at elevated temperatures are presented together with transmission electron microscopy investigations of changes in microstructure due to fatigue loading. Relation between cyclic behavior and microstructure evolution is discussed.

2. Experiment

The chemical composition of the Eurofer 97 steel in is wt. % is. 9Cr, 0.2V, 1W, 0.47Mn, 0.12C. The ODS Eurofer was produced from the Eurofer 97 steel by atomization and subsequent powder metallurgy with addition of 0.3 % Y₂O₃. Thermal treatment was applied on both variants, for details see [1,2]. Fatigue tests were performed in symmetrical cycle with constant total strain amplitude at room temperature (RT), 550 °C (Eurofer 97 steel), 650 and 750 °C (ODS Eurofer steel). Gauge lengths of all specimens were polished mechanically and electrolytically. Transmission electron microscopy (TEM) investigations were performed using Philips CM12.

3. Results

Microstructure of as-received state of both variants of Eurofer steels were already described earlier [1,2]. Briefly, prior austenitic grains of size of 10-15 μm were observed in the Eurofer 97 steel. Majority of microstructure consists of equiaxed grains or subgrains having the size of about 0.7 μm; martensitic laths are rare. Grain boundaries are decorated by Cr and W-rich carbides. Microstructure of the ODS Eurofer contains approximately equiaxed grains of the same size (about 0.7 μm). Elongated remnants of martensitic laths were rarely detected. Chromium rich carbides are situated in the grain boundaries. Initial dislocation density is low. High resolution TEM dark field technique revealed homogeneous oxide particles distribution. Particles of size from 5 nm to 50 nm were detected.

![Fig. 1 Softening curves measured for both variants of the Eurofer steel. (a) RT; (b) elevated temperatures.](image)

Cyclic softening curves are plotted in Fig 1. The expected strengthening effect is obvious. Stress amplitude for given total strain amplitude is almost doubled at RT, see Fig 1a. On the other hand, undesirable cyclic softening was measured at RT in the ODS Eurofer too. Nevertheless, oxide dispersion reduced cyclic softening, especially under loading with low amplitudes. Cyclic softening curves measured at elevated temperatures are shown in Fig. 1b. The effect of oxide particles is stronger than at room temperature. Cyclic softening persists in the Eurofer 97 steel at 550 °C, while cyclic response of the ODS
Eurofer steel is rather stable at both temperatures 650 °C and 750 °C. Moreover, stress amplitude at the half life is even higher for the ODS Eurofer cycled at 650 °C than in the Eurofer 97 cycled at 550 °C. It indicates that operating temperature could be increased of about 100 °C with the similar strength.

Microstructure of the Eurofer 97 steel after cyclic loading at room temperature with total strain amplitude of 0.4 % is shown in Fig. 2a. Changes caused by cyclic plastic deformation can be summarized as follows: overall dislocation density decreased, large grains were fractionated into subgrains with misorientation up to 5 degree, grains/subgrains larger than 1.5 μm are present in microstructure and carbides are situated on grain boundaries. Similar changes but in lesser extent were detected in the ODS Eurofer steel. Dislocation density decreased, but some individual dislocations pinned by oxide dispersion were found. Some grains grew up to 1 μm, but majority of microstructure remained stable. An example of microstructure of the ODS Eurofer steel cycled at room temperature with total strain amplitude of 0.7% is shown in Fig. 2b.

Microstructural changes are more pronounced in both variants after cycling at elevated temperatures. The Eurofer 97 specimens were subjected to cyclic loading at 550 °C. An example of microstructure cycled with amplitude of 0.4 % is shown in Fig. 3a. The presence of grains with size of several micrometers was detected. Submicron grains/subgrains were not found, only carbides inside large grains resemble former grains. Obviously, not only low angle boundaries were destroyed but also some high angle boundaries disappeared due to mutual effect of higher temperature and cyclic plastic deformation. An example of microstructure of the ODS Eurofer cycled at 650 °C with total strain amplitude of 0.7 % is shown in Fig 3b. The main features are similar with the ones detected in the Eurofer 97 steel, but again in lesser extent. Majority of grains did not grow due to fatigue loading, nevertheless, some grains of size up
to 2 μm were revealed. Most of the grains were found to be dislocation free, some individual dislocations pinned by small oxide particles were documented. The situation is similar in specimens tested at 750 °C. Some grains grew up to 2.2 μm. All others features are the same as detected in specimens tested at 650 °C.

4. Discussion

Undesirable cyclic softening of FM steels (P91, P92, F82H, Eurofer 97 etc.) is described in literature very well [1, 3–5]. Recently, these steels were strengthened by fine oxide dispersion. It was expected that oxide dispersion will improve cyclic strength and remove cyclic softening. The first assumption was confirmed [2,6], but cyclic softening still persists in ODS Eurofer steel, although in lesser magnitude. The reason can be found in microstructure evolution. The initial microstructure of both steels has similar attributes: grain/subgrain size of about 0.7 μm, carbides situated at grain boundaries and similar dislocation density. Dislocation density decreases due to fatigue loading in both variants similarly. The main difference can be found in final grain/subgrain size. While grains of size of 1.5 μm after loading at RT and grains of several micrometers in diameter after loading at 550 °C were found in the Eurofer 97 steel, only grains smaller than 2 μm were found in microstructure of the ODS Eurofer steel after fatigue loading at any temperature. Moreover, dislocations pinned by oxide particles in the ODS Eurofer steel were found. It indicates that oxide dispersion also stabilizes grain/subrain boundaries and their destruction is more demanding than in case of the Eurofer 97 steel. On the other hand, oxide particles cannot completely prevent grain growth. Obviously, oxide particle dispersion itself does not guarantee stable cyclic behavior, the role of matrix must be taken in to account too.

5. Summary

The results of this work can be summarized as follows:

- Oxide dispersion strengthens material significantly and reduces cyclic softening at any testing temperature.
- Oxide dispersion stabilizes microstructure, it slows down grain/subgrain growth even during cycling at 750 °C.
- Oxide dispersion could increase usable temperature of the Eurofer steel of about 100 °C.

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