



Strengthening mechanisms of different oxide particles in 9Cr ODS steel at high temperatures

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ARTICLE INFO

Keywords:

Iron alloys
Stress relaxation measurements
Modelling/simulations
Plasticity

ABSTRACT

The influence of different oxide particles on deformation mechanisms of 9Cr ODS steels at temperatures 400–600 °C is tested. Two ODS variants with Al_2O_3 and Y_2O_3 dispersed particles respectively and one variant without particles have been prepared by powder metallurgy. The deformation mechanisms are investigated by the tensile and stress-relaxation test at temperatures 400 °C and 600 °C. The analysis is based on a comparison of athermal and thermal stress components of the flow stress, apparent activation volume and a strain rate hardening. Microstructural parameters are evaluated upon SEM and TEM observations. The contributions of different strengthening mechanisms are assessed using the Taylor, Hall-Petch, dispersed barrier and Orowan models. Alumina particles provide the highest strengthening which can be explained by the stress decrease around the particles induced by a high difference between Young's moduli of alumina and ferritic matrix. This stress field can trap the dislocation in the vicinity of particles. Due to this stress decrease, alumina particles provide strengthening even at 600 °C in contrast to yttria particles that lose their effect due to their overcoming by a non-conservative motion of dislocations.

1. Introduction

The oxide dispersion strengthened (ODS) steels were developed as a suitable material for high temperature applications especially in the chemical or nuclear industries [1–3]. Their exceptional strength at high temperatures arises from the presence of oxide particles that act as the obstacles for dislocations motion [4]. The most common type of oxide particles used nowadays is yttria (Y_2O_3). They are stable at high temperatures and yttria can be also used in nuclear applications. Nevertheless, an addition of other types of oxides into the microstructure has been studied in recent years. Hoffmann et al. [5] have performed a comparative study on 5 different oxide types (Y_2O_3 , MgO , La_2O_3 , Ce_2O_3 , ZrO_2) added into the (RAF) steel. The different oxides influence the microstructure (oxide particles distribution, grain size) which is then manifested in mechanical properties. Generally, the best strengthening is achieved by minimizing the oxide particle size and increasing the density and regularity of their spatial distribution [6–8]. Dispersed oxide particles also stabilize small grain size at high temperatures which is important for improving creep resistance. This was shown in the comparative study done by Li et al. [9] with Y_2O_3 , La_2O_3 and CeO_2 . Positive effect on microstructural stability and particle distribution have also complex oxides created by an addition of particular

element into ODS steel with yttrium. Such studies were performed with scandium [10], hafnium [11] or aluminium [12].

The mechanism of interaction between an individual particle and a dislocation is based on incoherency at a particle-matrix interface. Dislocation is not able to easily pass through a particle and is forced to bend around a particle. This effect causes a dragging of dislocation motion. However, non-conservative motion of dislocations is activated at higher temperatures (above 600 °C) which allows them to easily overcome those particles. Their strengthening effect therefore disappears [13]. The experiments performed within this study compare the strengthening effect of yttria and alumina particles. The alumina ones show higher strengthening that is observed even at 600 °C. Therefore another effect of particles on dislocation motion is suggested and analyzed as an objective of presented study. Oxide particles have different elastic constants than a ferritic matrix. Such difference induces an elastic stress field around particles that can act as additional obstacle to dislocation motion. This field is present even at high temperatures, therefore it can help in dislocations trapping. At room temperature, alumina has high Young's modulus about 350 GPa while yttria has only 160 GPa [14,15]. The matrix is 9Cr ferritic steel with Young's modulus around 210 GPa. Such high difference in elastic properties should show the stress field effect. The reference material used for particle

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strengthening comparison analysis is 9Cr steel without dispersed particles. The strengthening mechanisms at the micro level are analyzed using the proper phenomenological models and FEM analysis of particle-matrix interaction. The necessary experimental data are obtained from microstructural analysis and mechanical tests combining tensile and stress relaxations. These tests are used to estimate athermal part of flow stress, activation volume and strain rate sensitivity. Similar studies were performed for example by Ramar et al. [16] for ODS Eurofer, or by Kim et al. [17–19] for nanoclustered ODS steels.

2. Experimental procedure

2.1. Material

Three different variants of ferritic-martensitic steel of 9Cr Eurofer type have been used in the current study. One variant without dispersed particles (oxide free) and two with dispersed yttria (Y_2O_3) - (Y ODS) and alumina (Al_2O_3) - (Al ODS) particles. Materials have been prepared by powder metallurgy. Dense samples have been prepared from composite powders by spark plasma sintering in a vacuum. Samples are annealed in two steps: 1100 °C for 30 min and air cooled followed by heating up to 750 °C for 2 h and air cooled. The dispersed particles are introduced into material by an internal oxidation process. Yttrium and aluminum powder particles react with trapped oxygen and precipitates inside material. An amount of yttrium and aluminum is added in order to get 0.25 wt% of dispersed oxide particles. Detailed information about a procedure of steel preparation can be found in [20] (Table 1).

2.2. Mechanical tests

All materials were tested using the flat tensile specimens with the gage section of $14 \times 3 \times 1.5 \text{ mm}^3$. The specimens were designed to fit into the cylinder produced by SPS technique (30 mm diameter, 10 mm height). Four specimens were cut by wire-EDM from the central part of one cylinder in order to avoid surface areas that can be contaminated by the carbon SPS mold. A surface of specimens was polished by SiC papers of grit size 800–2500 and finished by a diamond paste of size 1 μm . Tensile and stress relaxation tests were performed with an initial strain rate of about 10^{-3} s^{-1} at temperatures of 400 °C and 600 °C respectively. There were four specimens tested (2 tensile tests and 2 relaxation tests) for each material and temperature (24 specimens in total). The tests were performed on the ZWICK Z50 screw driven machine with the MAYTEC mounted furnace with an air atmosphere. All tests were displacement controlled and deformation of the specimens was measured by the extensometer. The stress relaxation tests were performed at different stress levels by holding the crosshead position constant for 30 s. Two relaxations per one test were done at different stress levels. A relatively short time period of dwell was chosen to avoid possible creep processes that can be active at given temperature range.

2.3. Experimental data analysis

The deformation processes and particle dislocation interactions were analyzed using the results of the stress relaxation tests in the following way. The loading force versus displacement curves were converted into the true stress (σ) - true plastic strain rate ($\dot{\epsilon}_p$) curves using Lee and Hart equations [21].

$$\sigma = \frac{P[l_0 + (x - P/S)]}{A_0 l_0} \quad (1)$$

Table 1

Chemical composition of the reference oxide free variant of steel in wt%.

Fe	Cr	W	Mn	V	Ta	Ti
Balance	9.0	1.0	0.5	0.2	0.1	0.3

$$\dot{\epsilon}_p = -\frac{\dot{P}}{S[l_0 + (x - P/S)]} \quad (2)$$

where P is the load, $\dot{P}$ is the load decrease rate, S is the stiffness of the loading train - specimen and the loading machine, x is the position of the crosshead. The specimen dimensions were represented by its initial gage length l_0 and the cross section area A_0 .

The activation volume which can be described as the number of atoms that must be coherently and thermally activated to move a dislocation over a local obstacle [22] can be estimated by fitting the stress decrease during the relaxation period. The apparent activation volume V_A was estimated from the relaxation cycle by fitting it with a logarithmic law [23]:

$$\Delta\sigma = -\frac{kT}{V_A} \ln\left(1 + \frac{t}{c}\right) \quad (3)$$

where $\Delta\sigma$ is the stress decrease during the relaxation, k is the Boltzmann constant, T is the absolute temperature and c is the parameter that results from the fitting of $\Delta\sigma$ - t data.

Other estimated material property was the strain rate sensitivity described as the slope in the log-log stress-plastic strain rate plot:

$$m = \frac{\partial \log(\sigma)}{\partial \log(\dot{\epsilon}_p)} \quad (4)$$

The analysis of the strengthening processes was based on the decomposition of the true flow stress into two main components:

$$\sigma = \sigma_\mu + \sigma^* \quad (5)$$

where σ_μ is the internal (athermal) component which arises from the long range interactions of dislocations with obstacles and the thermal component σ^* which represents the short range interactions. Thermal stress depends on the plastic strain rate and temperature.

The internal component was estimated from the first cycle of the relaxation test. There were two methods for its determination used in this study. The first one is based on a power law (Orowan) relation between the stress rate and the stress level [24,25]:

$$-\dot{\sigma} = \alpha\alpha(\sigma - \sigma_\mu)^k \quad (6)$$

which gives after integration the equation:

$$\sigma = \sigma_\mu + [A(k-1)]^{1/(1-k)}(t + t_0)^{1/(1-k)} \quad (7)$$

This equation is fitted onto experimental time-stress data by variation of parameters σ_μ , A , k and t_0 .

The second method is based on the hyperbolic function that fits the relaxation curve and it can be written in the form [26]:

$$\sigma = (\sigma_\mu t + \delta\sigma_0)/(t + \delta) \quad (8)$$

where σ_0 is the stress at the beginning of the relaxation and δ is the fitting parameter.

3. Results

The microstructural analysis was performed to obtain average grain size and characteristics of the smallest dispersed particles (radius, spacing). The overall view of the microstructure of different variants is shown in SEM images in Fig. 1. These type of images were used for grain size estimate using intercept method. The images show that the oxide particles are distributed within the whole volume of material. There are large particles (radius > 50 nm) presented in all three materials. Those are carbides that are relics of a manufacturing process. They have only minor contribution to the material strengthening, therefore they are excluded from further analysis. The smallest particles with nanometer size have the strongest impact on material strengthening. Therefore the detailed study of these particles was performed on TEM images represented in Fig. 2. The particle size and spacing were estimated using ImageJ software for image analysis. The results of the

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