ATOM PROBE STUDY OF THE EFFECT OF THERMAL AGING ON THE NANOSTRUCTURE OF OXIDE DISPERSION-STRENGTHENED STEELS

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Abstract. In the present work, the effect of thermal aging on the nanostructure of three oxide dispersion-strengthened steels with different alloying systems: Eurofer ODS, 10Cr ODS and KP-3 ODS was investigated by atom probe tomography. The investigated steels were aged at 650°C for 500 and 1000 h. Nanoscale clusters enriched in Y, O and Cr, as well as in V, Ti and Al depending on the alloying system of the steel were found in all states. Investigation of the changes in the nanostructure of steels after thermal aging for 500 h showed a significant increase in the number density of clusters in all steels, while after 1000 h of aging their number density decreased in Eurofer ODS and 10Cr ODS, or remained at the same level in KP-3 ODS. At 500 h, the retention (10Cr ODS, KP-3 ODS) or increase (Eurofer ODS) of the average cluster size was also observed, while at 1000 h the average size was retained in Eurofer ODS and 10Cr ODS, or experienced a slight decrease in KP-3 ODS. Analysis of the nanostructure change showed first an increase in the number density of clusters (while maintaining or increasing the average size) in all steels during aging up to 500 h, corresponding to the nucleation stage of new clusters. After aging for 1000 h, a decrease in volume density was found, corresponding to the maturation stage. These regularities are also confirmed by analyzing the changes in the concentration of chemical elements in the matrix.

Keywords: *clusters, atom probe tomography, oxide dispersion-strengthened steel, thermal aging*

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INTRODUCTION

Oxide dispersion strengthened (ODS) alloys and steels demonstrate superior high-temperature tensile strength and creep resistance compared to traditional steels [1-3]. This factor makes ODS steels extremely attractive for applications both under elevated thermal loads (for example, as engine turbine materials [4]) and under combined thermal and radiation exposures (as materials for advanced nuclear and thermonuclear power plants [5, 6]). The enhanced heat resistance and radiation resistance characteristics of ODS steels are provided by the presence in their structure of a large number of uniformly distributed oxide particles [4, 7, 8]. Considering this feature, it is important to study the stability of dispersed inclusions under conditions typical for operation.

As a result of complementary studies using transmission electron microscopy (TEM), atom probe tomography (APT), small-angle neutron scattering (SANS), and small-angle X-ray scattering (SAXS), it was established that the matrix of ODS steels contains not only small oxide particles (<10 nm) but also nanosized clusters (<4 nm) [9-14]. Although clusters contribute less (compared to oxide particles) to the mechanical properties of ODS alloys in their initial state [15, 16], they can act as nucleation centers for oxide particles [17, 18], which is important for the overall stability of the nanostructure of ODS materials under operating conditions. It has been established that during irradiation, under the influence of thermally and radiation-induced diffusion, interaction between oxides and clusters occurs, leading to the nucleation of small oxides from clusters and, thus, to the stabilization of the oxide subsystem [19, 20]. Therefore, clusters play a key role in maintaining the properties of ODS steels under radiation loads. It is worth noting that studies of irradiation effects are typically conducted at temperatures within the range of ~500°C (see, e.g., [19, 20]), which is below the upper limit of the expected operating temperature range for ODS steels in advanced nuclear applications, where operation up to temperatures of 650–700°C is anticipated [21–23]. Considering the importance of clusters' influence on the stability of the entire ODS material system, it is necessary to examine the effect of elevated temperatures on the material properties.

The purpose of this work is to apply atom probe tomography to analyze the behavior of ODS steels during thermal aging. For the study, a temperature of 650°C was selected, corresponding to the upper range of operating temperatures for ODS steels in advanced nuclear power plants.

MATERIALS AND METHODS

The materials studied were developed at the Karlsruhe Institute of Technology (KIT, Germany) – Eurofer ODS [24, 25], Kyoto University (Japan) – KP-3 ODS [26, 27], and the Korea Atomic Energy Research Institute (KAERI, Republic of Korea) – 10Cr ODS [28, 29]. All ODS steels were produced by mechanical alloying of metal powders and Y_2O_3 powder. The nominal compositions of the studied ODS steels are presented in Table 1.

Table 1. Chemical composition of the investigated ODS steels, at.% (balance by Fe)

Material	Mo	Al	Ni	Mn	Cr	W	Y	O	Ti	V	C	N	Ar	Si
Eurofer ODS	–	–	0.02	0.39	9.81	0.34	0.13	0.34	–	0.22	0.40	0.21	–	0.06
10 Cr ODS	0.57	–	–	0.50	10.64	–	0.17	0.17	0.29	0.11	0.60	0.02	0.01	–
KP-3 ODS	–	6.40	–	–	13.82	0.55	0.16	0.37	0.18	–	0.21	–	–	–

Eurofer ODS steel is alloyed with V, 10Cr ODS contains V and Ti, and KP-3 ODS steel is alloyed with Ti and Al. The content of yttrium in all steels is in the range of 0.13-0.17 at.%, while the oxygen content is represented in a fairly wide range from 0.17 to 0.37 at.%.

The initial states of these materials have already been previously studied by transmission electron microscopy and atom probe tomography [30]. This paper presents expanded APT data of the initial states of the studied steels, providing improved statistics of detected events.

To study thermal stability, samples of the investigated ODS steels were aged in a high-temperature electric vacuum furnace TVF-1200X. The heating time from room temperature (21°C) to 650°C was 90 minutes. The samples were kept at 650°C for 500 and 1000 h in vacuum of $5 \cdot 10^{-5}$ Torr. After stopping the heating, the cooling time in vacuum of $5 \cdot 10^{-5}$ Torr was 6 h.

The study of nanoscale precipitates in materials was conducted using atom probe tomography (APT) with picosecond laser evaporation on the PAZL-3D microscope [31]. Low temperature (40-50 K) combined with ultra-high vacuum and high laser energy (0.1-1.2 μJ) provided reduction of background noise. The laser source delivered pulses with a duration of 10 ps at a wavelength of 355 nm with a frequency of 100 kHz [32]. Evaporation ranged from 5 to 50 atoms per 1000 laser pulses, and the laser power was adjusted so that the ratio of Fe^{++}/Fe^{+} was in the range from 100 to 1000 relative units.

For atom probe tomography sample preparation, bars with dimensions of $0.3 \times 0.3 \times 10$ mm^3 were cut from the initial blanks. Further thinning of the samples was carried out using standard electrochemical polishing methods to form a sample tip with a radius of curvature of 15-50 nm. The quality of the obtained samples was controlled on a JEOL 1200 EX transmission electron microscope.

APT data analysis included mass spectrum identification, reconstruction and analysis of the three-dimensional distribution of chemical elements in the samples using KVANTM-3D software [33]. For each state, at least two volumes with dimensions of $30 \times 30 \times 500$ nm^3 were obtained.

RESULTS

APT analysis of the initial state of ODS steels revealed a significant number of nanoclusters with sizes in the range of 2-4 nm, enriched in Cr, Y, O, as well as Ti, V and Al, depending on the alloying system [30]. In Eurofer ODS steel, which does not contain Ti, the clusters contain a significant amount of vanadium. In 10Cr ODS steel, containing V and Ti in relatively similar concentrations, the clusters are predominantly enriched in Ti. In KP-3 ODS steel, containing ~30 times more Al compared to Ti, the clusters are nevertheless predominantly enriched in Ti.

After thermal aging at 650°C, clusters enriched in the same elements as the clusters in the initial states were found in the ODS steels. During aging for 500 hours in Eurofer ODS, the average cluster size increases by 2 times to 4 ± 1 nm, and their volume density increases by 1.5 times to 48×10^{22} m^{-3}. During thermal aging for 1000 h, the average cluster size remains unchanged, while the volume density decreases by a factor of 1.3 to 37×10^{22} m^{-3}. Atomic maps of chemical element distribution in the investigated volume of Eurofer ODS steel after thermal aging are presented in Fig. 1 , 2 , and cluster size distributions (normalized to volume density) are shown in Fig. 3 . For comparison, the cluster size distribution in the initial state is also presented in Fig. 3 .

Fig. 1. Atomic maps of chemical element distribution in the investigated volume of Eurofer ODS steel after aging at 650°C for 500 h.

Fig. 2. Atomic maps of chemical element distribution in the investigated volume of Eurofer ODS steel after aging at 650°C for 1000 h.

Fig. 3. Cluster size distributions in Eurofer ODS steel in the initial state, after thermal aging at 650°C for 500 h and 1000 h.

During thermal aging for 500 h at 650°C in 10Cr ODS, the average cluster size remains unchanged (4 ± 1 nm), while their volume density increases by a factor of 1.3 to 21×10^{22} m^{-3}. During thermal aging for 1000 h, the average cluster size remains unchanged and the volume density decreases to the value obtained in the initial state (within the scatter range). Atomic maps of chemical element distribution in 10Cr ODS steel after thermal aging are presented in Fig. 4 -5 , and cluster size distributions are shown in Fig. 6 . For comparison, the cluster size distribution in the initial state is also presented in Fig. 6 .

Fig. 4. Atomic maps of chemical element distribution in the investigated volume of 10Cr ODS steel after aging at 650°C for 500 h.

Fig. 5. Atomic maps of chemical element distribution in the investigated volume of 10Cr ODS steel after aging at 650°C for 1000 h.

Fig. 6. Cluster size distributions in 10Cr ODS steel in the initial state, after thermal aging at 650°C for 500 h and 1000 h.

Fig. 7. Atomic maps of chemical elements distribution in the investigated volume of KP-3 ODS steel after aging at 650°C for 500 h.

Fig. 8. Atomic maps of chemical elements distribution in the investigated volume of KP-3 ODS steel after aging at 650°C for 1000 h.

Fig. 9 . Cluster size distributions in KP-3 ODS steel in the initial state, after thermal aging at 650°C for 500 h and 1000 h.

During thermal aging for 500 h at 650°C in KP-3 ODS, the average cluster size remains within 4 ± 1 nm, while their volume density increases by 1.3 times to 60×10^{22} m^{-3}. During thermal aging for 1000 h, the average cluster size decreases to 3 ± 1 nm, and the volume density slightly increases to 62×10^{22} m^{-3} (within the scatter range). Atomic maps of chemical elements distribution in KP-3 ODS steel after thermal aging are presented in Fig. 7 , 8 , cluster size distributions, including in the initial state, are shown in Fig. 9 .

Table 2. Characteristic sizes and volume density of clusters detected using APT in ODS steels after thermal aging at 650°C for 500 and 1000 h (standard deviations are given, statistical error in volume density is determined by the number of detected clusters)

Material	Cluster type	Average cluster size, nm			Volume density of clusters, 10^{22} m^{-3}		
		Initial state	650°C, 500 h	650°C, 1000 h	Initial state	650°C, 500 h	650°C, 1000 h
Eurofer ODS	Cr–Y–O-V	2 ± 1	4 ± 1	4 ± 1	32 ± 5	48 ± 3	37 ± 5
10Cr ODS	Cr–Y–O–Ti	4 ± 1	4 ± 1	4 ± 1	16 ± 2	21 ± 2	15 ± 2
KP-3 ODS	Cr–Y–O–Ti–Al	4± 1	4 ± 1	3 ± 1	45 ± 4	60 ± 3	62 ± 5

Table 2 presents a comparison of the quantitative characteristics of clusters in the initial state and after thermal aging at 650°C for 500 h and 1000 h. A detailed comparison of cluster enrichment (difference between element concentration in clusters and its concentration in the matrix), as well as the matrix composition before and after thermal aging are presented in Tables 3 and 4 respectively. Negative enrichment in Table 3 corresponds to
lower element content in clusters compared to its content in the matrix. In Eurofer ODS, with increasing thermal aging time, the concentration of O and Y in the matrix and enrichment in clusters decreases. In 10Cr ODS, the concentration of O, Y, and Ti in the matrix decreases with increasing aging time, while in clusters, only O and Ti enrichment increases. In KP-3 ODS during aging, the concentration of O, Y, and Ti in the matrix increases, while in clusters, only O and Y enrichment increases.

Table 3. Enrichment of clusters in the studied ODS steels in the initial state and after thermal aging at 650 ° C for 500 and 1000 h, at.% (standard deviations are provided)

Material	Eurofer ODS			10Cr ODS			KP-3 ODS		
State / Element	Initial	500 h	1000 h	Initial	500 h	1000 h	Initial	500 h	1000 h
Fe	–43 ± 6	–36±3	–29 ± 10	–22±11	–36± 11	–24 ± 6	–20 ± 5	–24± 7	–31 ± 5
Cr	10 ± 1	6 ± 3	7 ± 4	4±2	4± 2	3± 2	3 ± 2	3± 2	7 ± 3
Y	12 ± 3	4 ± 2	4 ± 1	3± 2	3± 1	3 ± 2	2 ± 1	4 ± 1	4 ± 2
O	11±2	9± 1	7 ± 1	7± 4	16± 4	9 ± 3	8 ± 2	12 ± 1	12 ± 2
Ti	–	–	–	6 ± 3	10± 4	8 ± 3	7 ± 2	9 ± 1	7 ± 3
V	8± 2	13±5	7 ± 5	0.8±0.4	1± 1	0.6± 0.4	–	–	–
Al	–	–	–	–	–	–	0.2 ± 0.2	0.3± 0.1	0.8 ± 0.8

Table 4 . Average concentrations of chemical elements in the matrix before and after thermal aging at 650 ° C for 500 and 1000 h, at.% (standard deviations are provided)

Material	Eurofer ODS			10Cr ODS			KP-3 ODS		
State / Element	Initial	500 h	1000 h	Initial	500 h	1000 h	Initial	500 h	1000 h
Fe	89 ± 5	90 ± 1	91 ± 1	90 ± 1	91 ± 2	92 ± 1	82 ± 2	84 ± 1	86 ± 1
Cr	10 ± 1	9 ± 2	9 ± 1	8 ± 1	8 ± 1	7 ± 1	17 ± 2	15 ± 1	14 ± 1
Y	0.1 ± 0.1	0.02 ± 0.01	0.01 ± 0.01	0.2 ± 0.1	0.07 ± 0.07	0.04 ± 0.01	0.02 ± 0.02	0.07±0.01	0.05 ± 0.04

O	0.3 ± 0.1	0.1 ± 0.1	0.04 ± 0.01	0.1 ± 0,1	0.2 ± 0.2	0.01 ± 0.01	0.01 ± 0.01	0.1 ± 0.1	0.2 ± 0.1
Ti	–	–	–	0.07 ± 0.01	0.06 ± 0.06	0.02 ± 0.02	0.01 ± 0.01	0.07 ± 0.06	0.07 ± 0.01
V	0.2 ± 0.1	0.2 ± 0.1	0.1 ± 0.1	0.1 ± 0.1	–	–	–	–	–
C	–	0.06 ± 0.01	0.04 ± 0.02	0.02 ± 0.01	0.05 ± 0.03	0.1 ± 0.1	0.05 ± 0.03	0.02 ± 0.01	0.1 ± 0.1
N	–	0.07 ± 0.01	0.06 ± 0.01	0.01 ± 0.01	0.01 ± 0.01	0.01 ± 0.01	–	–	–
Mn	0.4 ± 0.1	0.3 ± 0.1	0.3 ± 0.1	0.7 ± 0.1	0.3 ± 0.3	0.4 ± 0.1	–	–	–
Mo	–	–	–	0.4 ± 0.1	0.4 ± 0.2	0.2 ± 0.1	–	–	–
W	0.3 ± 0.1	0.03 ± 0.01	0.05 ± 0.02	–	–	–	0.3 ± 0.1	0.04 ± 0.01	0.1 ± 0.1
Al	–	–	–	–	–	–	0.06 ± 0.01	0.03 ± 0.01	0.06 ± 0.01

DISCUSSION

Atom probe tomography investigation revealed significant changes in the cluster subsystem during thermal aging. Changes in the average size and volume densities of clusters with increasing thermal aging time from the initial state to 1000 h at 650°C are presented in Fig. 10 and 11 respectively. In Eurofer ODS, a noticeable increase in the average cluster size from 2 ± 1 to 4 ± 1 nm is observed after aging for 500 h, followed by size preservation until the end of thermal aging. In 10Cr ODS, the size remains at ~4 ± 1 nm throughout the entire range of aging times, while in KP-3 ODS, the average size is stable (~4 ± 1 nm) up to 500 h and slightly decreases (to 3 ± 1) when the aging time increases to 1000 h. It should be noted that in all three steels, after reaching 1000 h of aging (Fig. 3 , 6 and 9), large clusters (> 6–9 nm) that were still present after 500 h are no longer observed.

Fig. 10. Dependence of the average cluster size on the degree of thermal aging.

Fig. 11. Dependence of the volume density of clusters on the degree of thermal aging.

Based on changes in the quantitative and qualitative characteristics of clusters and concentrations of chemical elements in the matrix of the studied ODS steels (table 2 and 4), conclusions can be drawn regarding the nature of changes in the cluster subsystem. For this, we will estimate the volume of material contained in clusters, $4/3\ \pi R^3 \times N$, where R and N are the radius and volume density of clusters, respectively. As a result of aging at 650°C, 500 h in Eurofer ODS, the volume of material in clusters significantly increases compared to the initial state, which is also accompanied by a characteristic decrease in the amount of Y and O in the matrix, corresponding to the stage of nucleation and growth of clusters from the supersaturated matrix solution. Similarly, in 10Cr ODS, an increase in the total volume of clusters is observed, with a decrease in the content of Y and Ti in the matrix, which corresponds to the trend characteristic of the nucleation and growth stage of clusters. In KP-3 ODS, an increase in the total volume of clusters is also observed, but with an increase in the content of Y, O, and Ti in the matrix, which is possible when, in addition to nucleation and growth of clusters, these elements are released due to dissolution of oxide particles. After aging for 1000 h in Eurofer ODS, 10Cr ODS, and KP-3 ODS, a decrease in the volume of clusters is observed compared to 500 h with a further decrease in the content of corresponding elements in the matrix. The decrease in the volume of clusters with an increase in aging time from 500 to 1000 h is presumably associated with the transformation of large clusters into oxide particles.

According to the simulation results from work [34], the critical nucleation radius for Y–O and Y–Ti–O type clusters is within 2 nm. After that, according to [35], coalescence (or Ostwald ripening in English literature) plays an active role [1, 8, 13, 36]. This pattern is fully consistent with the results obtained in the present work for Eurofer ODS and 10Cr ODS steels. In Eurofer ODS (Y–O clusters) after aging for 500 hours at 650°C, a sharp increase in the average size (up to 4 ± 1 nm) and volume density of clusters (by 1.5 times) is observed, which corresponds to the nucleation stage, while during aging for 1000 hours, the average size is maintained with a decrease in volume density (by 1.3 times), indicating the transition of larger clusters to oxide particles. In 10Cr ODS, the average size of Y–Ti–O clusters is stable (4 ± 1 nm), while the volume density first increases (by 1.3 times at 500 hours) and then decreases (by 1.3 times at 1000 hours), which is also well described by a model including stages of nucleation, growth, and subsequent coalescence [34, 35]. The behavior of KP-3 ODS steel differs: at 500 hours of aging, the average size value remains within 4 ± 1 nm with an increase in volume density (by 1.3 times), but then at 1000 hours decreases to (3 ± 1 nm) while maintaining volume density. This change is presumably associated with the transformation of larger clusters into oxides. Verification of this statement requires additional TEM research.

CONCLUSION

An investigation was conducted using atom probe tomography methods on the restructuring of the nanostructure of oxide dispersion strengthened steels Eurofer ODS, 10Cr ODS, and KP-3 ODS with different alloying systems as a result of thermal aging at 650°C for 500 and 1000 hours.

After aging of the ODS steels for 500 h, the following were found: Cr–Y–O clusters with sizes of ~4 nm and volume density of $\sim 48 \times 10^{22}$ m^{-3} in Eurofer ODS, Cr–Y–O–Ti clusters with an average size of ~4 nm and volume density of 21×10^{22} m^{-3} in 10Cr ODS steel, and Cr–Y–O–Ti clusters with sizes of ~4 nm and volume density of 60×10^{22} m^{-3} in KP-3 ODS steel. After aging for 1000 h, the type of clusters did not change, and their characteristics were: average size of ~4 nm and volume density of $\sim 37 \times 10^{22}$ m^{-3} in Eurofer ODS, size of ~4 nm and volume density of 15×10^{22} m^{-3} in 10Cr ODS steel, and size of ~3 nm and volume density of 62×10^{22} m^{-3} in KP-3 ODS steel.

Comparison of the obtained data after thermal aging with the data for the initial state demonstrates an increase in the volume density of clusters (while maintaining or increasing their average size) in all steels during aging for 500 h, corresponding to the stage of cluster nucleation. While after aging for 1000 h, a decrease in volume density was found, which corresponds to the coalescence stage. These patterns are also confirmed by the analysis of changes in the concentration of chemical elements in the matrix.

Changes in volume density, average sizes, and chemical composition of clusters, as well as the matrix, indicate significant interaction of clusters with another phase under thermal aging conditions at 650°C for up to 1000 h. Oxide particles may act as this phase.

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CONFLICT OF INTERESTS

The authors of this work declare that they have no conflict of interest.

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