THE CHEMISTRY ON A SUBNANOMETER SCALE OF RADIATION-INDUCED PRECIPITATION AND SEGREGATION IN FAST-NEUTRON IRRADIATED TUNGSTEN-RHENIUM ALLOYS

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ABSTRACT

The phenomena of radiation-induced precipitation and segregation have been investigated in W-10 at.% Re and W-25 at.% Re alloys, employing the atom-probe field-ion-microscope technique. The W-10 at.% Re alloy is subsaturated with respect to the solvus line of the primary solid solution (β phase), while the W-25 at.% Re alloy is supersaturated with respect to the same solvus line. The specimens had been irradiated in the Experimental Breeder Reactor II to a fast-neutron fluence of $\sim 4\times10^{22}$ neutrons cm^{-2} (E>0.1 MeV) at 575, 625 and 675°C. This corresponds to 8.6 dpa and an average displacement rate, for the two year irradiation time, of 1.4×10^{-7} dpa s^{-1}. The results of the present investigation show a very significant alteration of the microstructure of both alloys as a result of the fast-neutron irradiation. In the case of the W-10 at.% Re alloy coherent, semicoherent and possibly incoherent precipitates with the composition ~WRe and a disc-shaped morphology -- one or two atomic planes thick -- were detected at a number density of $\sim 10^{16}$ cm^{-3}, and a mean diameter of ~57 Å. For the W-25 at.% Re alloy coherent, semicoherent and incoherent precipitates with the composition $\sim WRe_3$ were detected; the precipitate's number density is $\sim 10^{17}$ cm^{-3} with a mean diameter of 40 Å. None of the ~WRe precipitates or the $\sim WRe_3$ coherent precipitates were associated with either line or planar defects or with any impurity atoms. Therefore, a true homogeneous radiation-induced precipitation occurs in these alloys. The semicoherent WRe_3 precipitates were associated with ^4He atoms; that is, these precipitates may have been heterogeneously nucleated. In the W-25 at.% Re alloy a two dimensional WRe_3 phase has been observed at a grain boundary. A physical argument is presented for the nucleation of WRe or WRe_3 precipitates in the vicinity of displacement cascades produced by primary knock-on atoms. It is suggested that in both cases the first step in the nucleation of a precipitate is due to the formation of tightly-bound mobile mixed dumbbells which react to form an immobile di-rhenium cluster. Possible sequences of point-defect reactions which can lead to either WRe or WRe_3 cluster are detailed. The further growth of a cluster (WRe or WRe_3) into a precipitate is most likely driven by the irreversible vacancy: self-interstitial atom annihilation reaction, as suggested recently by Cauvin and Martin.[24] Point-defect mechanisms for all the other observations are also discussed.

INTRODUCTION

Over the last few years there has been a rapid growth of interest in the phenomena of radiation-induced (as opposed to accelerated) segregation and precipitation.[1-4] Different types of irradiation -- electrons, ions or neutrons -- can induce significant segregation of alloying elements either toward or away from grain boundaries, voids or free surfaces. Radiation can also cause the heterogenous or homogeneous precipitation of a phase in subsaturated solid solutions and it can alter the phase stability of alloys.[1-4] Radiation-induced segregation and precipitation are of paramount technological importance, since they play a crucial role in the nucleation and growth of voids and have a strong effect on the physical properties of metals alloys used in the fuel cladding and core structure of the fast-breeder reactor, as well as in the materials used for the first wall of fusion reactors. These phenomena are also of considerable fundamental interest.

The study of W(Re) alloys is of technological importance, as they are used in thermocouples for the measurement of temperature in nuclear reactors. As a result of an exposure to a fast-neutron flux the decalibration of W(Re) thermocouples occurs.[5,6] The alloys W-10 at.% Re and W-25 at.% Re are of particular interest in understanding the phenomenon of radiation-induced precipitation, as the former alloy is subsaturated with respect to the solvus line of the primary solid solution (β phase), while the latter alloy is supersaturated with respect to this solvus line -- it is in the β plus σ phase field.[7,8] Sikka and Moteff[9] and Williams et al.[10] have identified the crystal structure of radiation-induced precipitates in fast-neutron irradiated W-25 at.% Re alloys using transmission electron microscopy -- for specimens which had been irradiated at 1100°C and higher -- and it corresponds to the χ-phase which has the composition WRe_3. Williams et al.[10] also investigated fast-neutron irradiated W-5 at.% Re and W-11 at.% Re alloys. For specimens which had been irradiated at 1100°C and above all the precipitates analyzed by electron diffraction were consistent with the χ-phase crystal structure. Whereas Williams et al.[10] were unable to obtain interpretable electron diffraction patterns from any of the specimens which had been fast-neutron irradiated at 900°C or lower.

In this short summary paper we present the results of an extensive atom-probe field-ion microscope (FIM) study of radiation-induced precipitation and segregation in fast-neutron irradiated W-10 at.% Re and W-25 at.% Re alloys.[11,12] Our atom probe FIM allows us to determine the chemical identity of all the elements in the periodic table.[13-16] In addition, the atom-probe FIM has a lateral spatial resolution, for chemistry, of a few-tenths of a nanometer and a depth resolution which is determined by the interplanar spacing of the region being analyzed.

We found very significant alterations of the microstructure between 575 to 675°C. In the case of the W-10 at.% Re alloy precipitates with the composition ∼WRe (σ phase) were detected at a number density of $\sim 10^{16} cm^{-3}$. They were not associated with linear or planar defects or with any impurity atoms; i.e., a true homogeneous radiation-induced precipitation occurs in this alloy. Coherent, semicoherent and incoherent precipitates were detected. For the W-25 at.% Re alloy coherent, semicoherent and incoherent precipitates with the composition $\sim WRe_3$

(χ phase) were detected at a number density $\sim 10^{17}cm^{-3}$. The $\sim WRe_3$ coherent precipitates were not associated with either line or planar defects, or with any impurity atoms. This strongly suggests that the coherent WRe_3 precipitates were a result of a homogeneous radiation-induced process. The semicoherent and incoherent $\sim WRe_3$ precipitates were found to be associated with ^4He atoms; i.e., they may have been heterogeneously nucleated. In addition we found evidence for a two-dimensional $\sim WRe_3$ phase at a grain boundary; this phase is the result of a radiation-induced segregation process.

EXPERIMENTAL DETAILS

Wire specimens of W(Re) alloys were irradiated to a fast neutron-fluence of $\sim 4x10^{22}$ neutrons cm^{-2} (E>0.1 MeV) at elevated temperatures (575, 625 and 675°C) in Experimental Breeder Reactor II (EBR-II) at Richland, Washington. This corresponds to 8.6 dpa for row 7 of EBR-II. Hence, the average displacement rate for the two year irradiation time is $1.4x10^{-7}$ dpa s^{-1}.

The wire specimens were electroetched into sharply-pointed FIM specimens. Next the specimens were analyzed chemically by the atom-probe technique at an ambient pressure of $\sim 4x10^{-10}$ Torr, with the specimens maintained at 45 K. A pulse fraction (f) of 0.15 was used for all the analyses. The quantity f is the ratio of the pulse voltage to the steady-state dc voltage. A constant pulse frequency of 60 Hz was employed. The average field-evaporation rate -- average number of ions evaporated per field-evaporation pulse -- was equal to 0.02 ions $pulse^{-1}$. Using these experimental conditions we were able to obtain good agreement between the nominal Re concentration, and the Re concentration as determined by the atom-probe technique for unirradiated alloys. These experimental conditions were used in all of our chemical analyses.

The specimens were imaged employing ^3He as an imaging gas. The reason for using ^3He, rather than ^4He gas, was to minimize the concentration of ^4He present in the atom probe and therefore, to make it possible to identify ^4He atoms which have had their origin in the neutron-irradiated specimens.

The basic mode of displaying the data in the present experiment is in the form of an integral profile. A Re integral profile is obtained by plotting the cumulative number of Re events versus the cumulative number of W plus Re events. The average slope of such a plot corresponds to the average Re concentration of the volume analyzed, since the cumulative number of all events detected is proportional to depth. In analyzing a particular precipitate the slope of an integral profile ($\langle c_{Re}^{ppt}\rangle_u$) is a lower limit to the actual Re concentration in a precipitate ($\langle c_{Re}^{ppt}\rangle^*$), as in most cases the dimensions of the analyzed cylinder are greater than the size of a precipitate. The superscript ppt stands for precipitate, the subscript u on the bracket means an uncorrected value and the superscript * implies a corrected value. The relationship between $\langle c_{Re}^{ppt}\rangle_u$ and $\langle c_{Re}^{ppt}\rangle^*$ for different possible precipitate morphologies is presented in Appendix A of Herschitz and Seidman.[11]

EXPERIMENTAL RESULTS: W-10 AT.% Re

Four radiation-induced precipitates were detected and analyzed, whereas no voids were found in the W-10 at.% Re alloy. The density of the radiation-induced precipitates is equal to $\sim 10^{16} cm^{-3}$; it was determined following the procedure used by Brenner and Seidman.[17]

The following summarizes the main experimental results:

(1) Coherent, semicoherent and possibly incoherent precipitates have been observed. The number density of precipitates is $\sim 10^{16} cm^{-3}$.

(2) The observed precipitates are disc shaped -- one or two atomic planes thick. And their mean diameter is ~ 57 Å.

(3) The composition of the radiation-induced precipitates corresponds to $\sim$WRe; that is, $\langle c_{Re}^{ppt} \rangle^* \underset{\sim}{\sim} 52$ at.% Re. This result indicates that the WRe precipitates in the W-10 at.% Re alloy are radiation resistant in the temperature range 575 to 675°C -- in the presence of a fast-neutron flux.

(4) The precipitates were not associated with either linear or planar defects, or with any impurity atoms; i.e. a true homogeneous radiation-induced precipitation occurs in this alloy.

(5) No voids were detected in this alloy. This indicates that the addition of 10 at.% Re to W suppresses void formation, as voids have been detected in pure tungsten -- which had been subjected to fast-neutron irradiation.

EXPERIMENTAL RESULTS: W-25 AT.% Re

Six precipitates, three voids, a grain boundary and a region immediately adjacent to a grain boundary were chemically analyzed by the atom probe technique.

The following summarizes the main experimental results for this alloy:

(1) Coherent, semicoherent and incoherent precipitates have been observed. Their number density is $\sim 10^{17} cm^{-3}$ and the mean diameter is ~ 40 Å.

(2) The precipitates observed have either a disc shaped or spherical morphology.

(3) The composition of the radiation-induced precipitates corresponds to $\sim WRe_3$; i.e., $\langle c_{Re}^{ppt} \rangle^*$ is approximately equal to 75 at.% Re.

(4) The coherent precipitates ($\sim WRe_3$) were not associated with either linear or planar defects or with any impurity atoms; i.e., a true homogeneous radiation-induced precipitation occurs in this alloy.

(5) The semicoherent and incoherent precipitates were associated with ^4He atoms; i.e., heterogeneous precipitation may have occured in this alloy.

(6) Voids at a number density of $\sim 10^{17} cm^{-3}$ and a mean diameter of 90 Å have been detected in this alloy. No significant Re enrichment or depletion at these voids had occured.

(7) Formation of a two-dimensional $\sim WRe_3$ phase has been observed at a grain boundary.

DISCUSSION

W-10 at.% Re alloy

The fact that the precipitates in the subsaturated alloy are not associated with either structural defects or with any impurity atoms indicates that a true homogeneous radiation-induced precipitation occurs in this alloy. Experimental evidence for homogeneous radiation-induced precipitation has been presented recently by Cauvin and Martin in the case of Al (Zn) alloys,[18,19] by Brager et al.[20] in the case of a 316 stainless steel, by Mukai and Mitchell for a Ni (Be) alloy,[21] and Kinoshita and Mitchell[22] and Wahi and Wollenberger[23] for Cu (Be) alloys. Theoretical treatments of this physical phenomenon have been considered by Cauvin and Martin[24] and Maydet and Russell;[25] the latter authors only considered the possibility of the nucleation of incoherent precipitates, whereas Cauvin and Martin[24] also considered coherent precipitates.

We now describe a possible sequence of plausible events which can result in the homogeneous nucleation of WRe (the σ phase) precipitates, in a subsaturated alloy which is subject to irradiation with fast neutrons.[11,12]

The primary source of radiation damage, in the case of fast neutrons, is the displacement cascade. Each displacement cascade is created by a primary knock-on atom (PKA) with a mean recoil energy of 4 keV. In the case of pure tungsten it is known from FIM experiments that a displacement cascade, created at 15 K, consists of a vacancy-rich core ($\sim$2 to 30 at.%) surrounded by a distribution of self-interstitial atoms (SIAs), which is created by the replacement collision sequence mechanism.[26,27] The concentration of SIAs on the periphery of a displacement cascade can be as high as $\sim$1 to 3 at.%. Since the radiation damage is highly localized in the displacement cascade -- the point defect supersaturation in between the displacment cascades is initially negligible -- it is probable that the nucleation of a WRe precipitate occurs in its vicinity.[11] The absolute efficiency of this nucleation process is as low as the final density of radiation - induced WRe precipitates is $\sim 10^{16} cm^{-3}$ -- which is significantly less than the number density of PKAs that produce displacement cascades.[11]

Employing the known properties of point defects in W and W(Re) alloys it can be demonstrated that plausible first steps in the nucleation of a WRe (σ phase) precipitate involve the migration of tungsten SIAs to Re atoms to form mobile mixed dumbbells -- in the immediate vicinity of a displacement cascade -- which in turn react to form an immobile di-Re cluster.[11] The di-Re cluster can then grow by the accretion of mixed dumbbells, and pure tungsten or rhenium SIAs. Specifically, the formation of a WRe cluster is envisaged to occur via the following possible reactions: (a) two mixed dumbbells react to form an immobile di-Re cluster; (b) the di-Re cluster reacts with a pure tungsten SIA to form a WRe_2 cluster; and (c) the WRe_2 cluster reacts with a second tungsten SIA to form W_2Re_2 (or 2WRe) cluster. During the course of the two year irradiation the displacement cascades dissolve slowly (see Appendix B of Herschitz and Seidman[11]) and they provide the vacancies which can result in the shrinkage of clusters. Recent experiments by Averback and Ehrhart[28] on Ni-1 at.% Si also suggest strongly that point defect clustering and trapping reactions occur in the vicinity of displacement cascades.

Further specific details of the growth or shrinkage of a cluster are difficult to state, but they can be rationalized in terms of the Cauvin-Martin model[24] for radiation-induced metastability. The physical basis of the Cauvin-Martin model[24] is that the irreversible vacancy-SIA annihilation reaction drives solute clusters towards a larger solute content and hence to precipitation.

A plausible mechanism for the suppression of void swelling, in this alloy, involves the dominance of vacancy: SIA recombination over the destruction of these point defects at a biased sink -- the dislocation. This is possible, in particular, by the recombination of vacancies with SIAs which are trapped in immobile clusters involving SIAs and rhenium atoms. This strong recombination process prevents the accumulation of a sufficient number of vacancies for the nucleation and growth of voids. This mechanism for the suppression of voids is consistent with the mechanism suggested for the homogeneous nucleation of WRe (σ phase) precipitates.

W-25 at.% Re alloy

A very striking observation is the detection of coherent precipitates with the composition WRe_3, in the W-25 at.% Re alloy, which are not associated with either structural defects or impurity atoms. The latter observation strongly suggests that they were homogeneously nucleated. The basic problem is to explain the radiation-induced precipitation of WRe_3 if the χ phase is not in thermal equilibrium with the primary (β) solid solution between 575 to 675°C.

The χ phase may also be nucleated by the homogeneous nucleation mechanism suggested in the previous subsection for the σ phase in the vicinity of displacement cascades. The formation of a WRe_3 cluster is envisaged to occur via the following reactions: (a) two mixed dumbbells react to form an immobile di-Re cluster; (b) the di-Re cluster reacts with another mixed dumbbell to form a Re_3 cluster; and (c) the Re_3 cluster reacts with a pure tungsten SIA to form a WRe_3 cluster. Once again the further growth (or shrinkage) of this elementary cluster can be understood to occur via the Cauvin-Martin model[24] for radiation-induced metastability.

A necessary condition for the formation of a large number density of WRe_3 precipitates is that the nucleation current of these immobile WRe_3 clusters be considerably greater than the nucleation current of immobile WRe clusters -- in the W-25 at.% Re alloy. In the previous subsection we suggested three point defect reactions which can lead to the formation of a W_2Re_2 (or 2WRe) cluster. If it is assumed that these nucleation reactions, as well as the three reactions postulated above for the nucleation of a WRe_3 cluster, are in detailed balance then it is readily shown that

$$c_{WRe_3} \propto c_{W-W} c^3_{Ii}$$

and

$$c_{W_2Re_2} \propto c^2_{W-W} c^2_{Ii} ;$$

where c_{W-W} is the concentration of tungsten SIAs and the constants of proportionality are the products of the rate constants of the individual point defect reactions that lead to WRe_3 of W_2Re_2 (2WRe). The above equations show that c_{WRe_3} should be greater than $c_{W_2Re_2}$. This is because the value of c_{Ii} grows more rapidly -- in the direct vicinity of a displacement cascade -- in the supersaturated W-25 at.% Re alloy than in the W-10 at.% Re alloy; note that c_{W-W} is initially the same in both alloys and is in the range 10^{-2} to $3x10^{-2}$ at. fr.

This model for the homogeneous nucleation of WRe_3 precipitates, from a supersaturated W-25 at.% Re alloy, shows qualitatively that the nucleation current of immobile WRe_3 clusters is greater that of W_2Re_2 (2WRe) clusters. However, the model does not explain why in the subsaturated W-10 at.% Re alloy the Re concentration of the precipitates stops at ∿50 at.% Re. This question stands as an unsolved problem at present.

Another very interesting observation is the detection of ^{4}He atoms inside semicoherent or coherent participates. The detection of ^{4}He atoms in this alloy is at first glance somewhat surprising, as the cross section for ^{4}He production in pure tungsten is quite small. The cross section for the production of ^{4}He atoms on rhenium atoms is not available, however L.R. Greenwood (Argonne National Laboratory, private communication) estimates that the ^{4}He production rates on Re and W should be quite similar. Thus, for a fluence of 8.6 dpa the estimated ^{4}He concentration is ∿$4x10^{-3}$ appm in W-25 at.% Re; this assumes the displacement threshhold energy of a Re atom is identical to that of a W atom -- 52 eV.[29] This suggests that the ^{4}He atoms were most likely produced on the impurity atoms present in this alloy. The elements B, C, N, O and S all have rather large cross sections for the production of ^{4}He atoms (L.R. Greenwood, private communication). The absence of ^{4}He atoms in precipitates in the W-10 at.% Re alloy[11] may simply be a result of a lower level of impurity atoms in this particular alloy.

Herschitz and Seidman[12] discuss four possible mechanisms which can result in the detection of ^{4}He atoms in the WRe_3 precipitates. Two of the mechanisms imply that the WRe_3 precipitates were heterogeneously nucleated and two that they were homogeneously nucleated. There is no obvious way to distinguish among these four mechanisms. Hence, we are left with the distinct possibility that the semi or incoherent WRe_3 precipitates were heterogeneously nucleated.

In the voids analyzed by the atom probe technique we were unable to detect ^{4}He atoms. The specimen temperature was 45 K during the chemical analyses; hence, we can rule out the possibility of ^{4}He atoms diffusing out of the voids as they were dissected, since the measured mobility of ^{4}He atoms at 45 K is extremely small.[30-32] The most likely reason we were unable to detect ^{4}He atoms in the voids was simply that the volume fraction of the void analyzed was small -- typically much less than 0.1, and since the absolute number of ^{4}He atoms per void is not expected to be very large, the probability of detecting one ^{4}He event is very small.

The corrected Re concentration in a grain boundary was determined to be ∿75 at.% Re. This value corresponds to WRe_3 (χ phase). The grain boundary Re concentration was found to fall to the bulk value (25 at.% Re) in ∿4 Å. Thus, the χ phase forms along a grain boundary and we have an example of a two-dimensional phase, which is the result of a non-equilibrium radiation-induced segregation process. The atomic mechanism for the formation of this phase can be explained in terms of the migration of mixed dumbbells to the grain boundary. This mechanism is consistent with our suggestion that the WRe_3 precipitates form in a W-25 at.% Re alloy, subject to a fast-neutron radiation field, as the result of a homogeneous nucleation process which involves mixed dumbbelis reacting to form an immobile di-Re cluster.

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