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# Precipitation in MA/ODS Ferritic Alloy MA957

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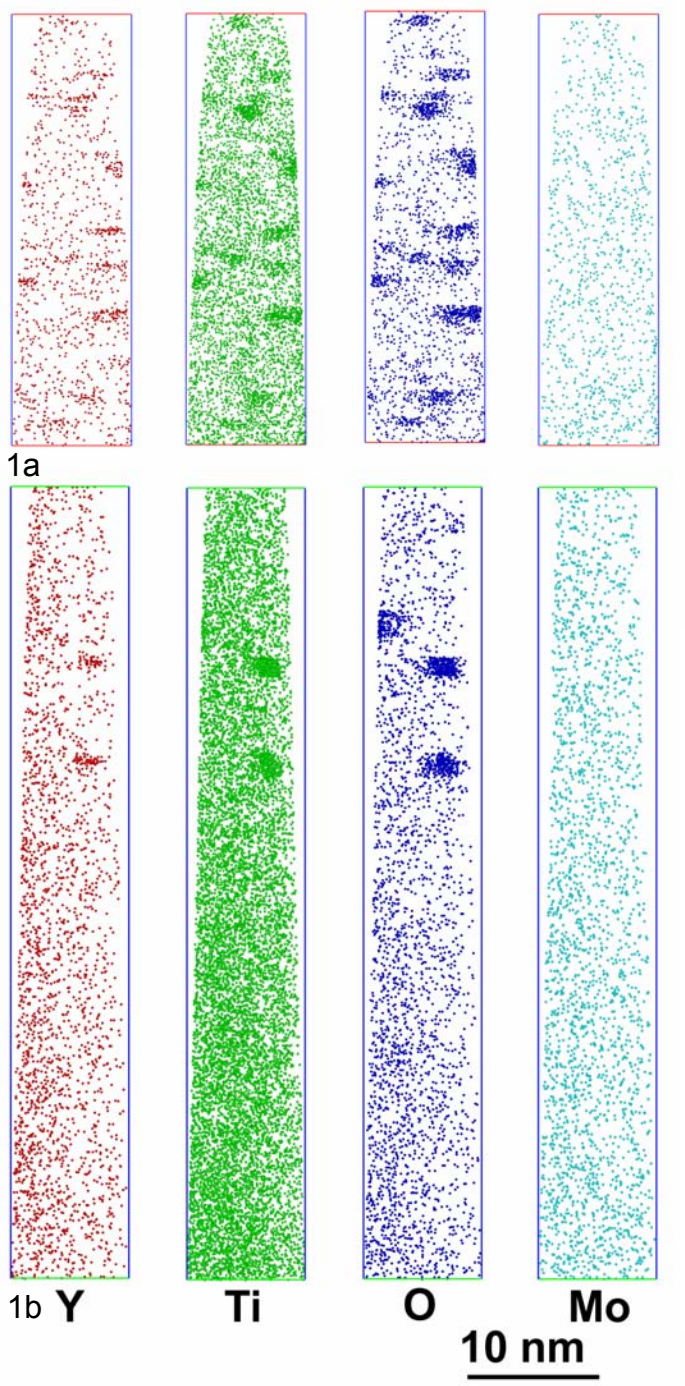
Some mechanically-alloyed (MA), oxide dispersion strengthened (ODS) ferritic alloys have exhibited dramatically improved high temperature creep properties compared to other ferritic alloys. These alloys are fabricated by ball milling powders of the prealloyed metal with an oxide powder. Atom probe tomography (APT) has revealed that some of these MA/ODS alloys [e.g., 12YWT] contain a high number density of Ti-, Y-, O-enriched particles that are stable to at least 1300°C (~85% $T_m$) [1]. In this paper, a commercial MA/ODS ferritic alloy MA957 is characterized by APT to determine if these ultrafine particles are a common feature of these MA/ODS ferritic alloys.

The commercial MA957 alloy used in this study had a nominal composition of Fe- 14 wt % Cr, 0.9% Ti, 0.3% Mo and 0.25% $Y_2O_3$ [Fe-14.8 at. % Cr, 0.17% Mo, 1.0% Ti, 0.13% Y and 0.19% O] and contained trace levels of Al, Mn, Si, B and C. Specimens were cut from an extruded tube. This alloy was characterized in the as-received state and after annealing for 1 h at 1300 °C. TEM revealed partial recovery of the dislocation structure but no recrystallization had occurred during the high temperature anneal.

The solute distribution in this MA957 alloy is shown in the atom maps in Fig. 1. A high number density (~2 x $10^{24}$ $m^{-3}$) of ultrafine Ti-, Y- and O-enriched particles are evident in the as-received condition, Fig. 1a. The number density of the particles decreased by an order of magnitude to ~2 x $10^{23}$ $m^{-3}$ after the 1 h at 1300°C anneal, Fig. 1b. The average Guinier radius of the particles, $r_G$, was determined with the use of the maximum separation method [2] to be 1.2 ± 0.4 and 1.7 ± 0.7 nm, respectively for the as-received and 1 h at 1300 °C conditions. These results indicate that some coarsening of the particles had occurred during the anneal. A representative atom map of a 2-nm-thick slice through the center of one of the particles in the annealed material is shown in Fig. 2. The Y atoms were predominantly found in the central region and surrounded with a Ti- and O-enriched shell, as apparent by the $TiO^{2+}$ ions. This differing solute distribution is also evident in the atom maps shown in Fig. 1. In both conditions, $r_G(Y)$ was approximately 90% of the overall $r_G$ value. The compositions of the individual particles were determined by the envelope method [2] with a grid spacing of 0.1 nm. The average compositions and the solute partitioning factors are given in Table 1. The oxygen content in the matrix was estimated to be <0.14 and <0.19 at. % O, respectively for the as-received and annealed conditions. These estimates are upper bounds due to the possible presence of $Mo^{3+}$ and $Ti^{3+}$ ions superimposing with the $TiO^{2+}$ and $O^+$ ions, respectively. These results are similar to previous APT studies of a 3 wt% W, Mo-free MA/ODS 12YWT ferritic alloy [1]. [3]

[1] D.J. Larson et al., Scripta Mater., 44 (2001) 359; M.K. Miller et al., Mat. Sci Eng. A. in press.
[2] M.K. Miller, *Atom Probe Tomography*, Kluwer Academic/ Plenum, New York, 2000.
[3] Research at the Oak Ridge National Laboratory SHaRE Collaborative Research Center was sponsored by the Laboratory Directed Research and Development Program and the Division of Materials Sciences and Engineering and the Office of Nuclear Energy. Science and Technology (I-NERI 2001-007-F), U.S. Department of Energy, under contract DE-AC05-00OR22725 with UT-Battelle, LLC.



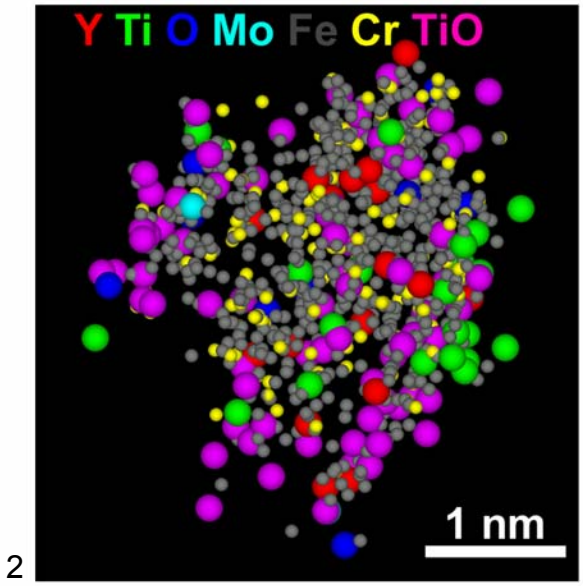




TABLE 1. Compositions and partitioning factors of the particles in the as-received and annealed conditions. The balance is Fe.

| at. % | As received | | 1 h at 1300 °C | |
|---|---|---|---|---|
| | $\overline{X}$ | PF | $\overline{X}$ | PF |
| Cr | 1.7±1.7 | 0.1 | 4.2±4.2 | 0.3 |
| Mo | 0.02±0.2 | 0.1 | 0.03±0.03 | 0.2 |
| Ti | 32.9±5.3 | 41 | 21.4±7.9 | 26 |
| Y | 15.4±7.3 | 91 | 8.9±5.8 | 52 |
| O | 39.9±6.9 | 285 | 47.8±23.3 | 294 |
| Al | 0.0 | 0.0 | 0.12±0.12 | 1.7 |
| Mn | 0.01±0.01 | 0.2 | 0.0 | 0.0 |
| B | 0.0 | 0.0 | 0.02±0.02 | 0.3 |
| C | 0.0 | 0.0 | 0.0 | 0.0 |
| Si | 0.0 | 0.0 | 0.05±0.05 | 0.6 |

FIG. 1. Atom maps of the solute distribution in MA957 in the a) as received condition and b) after annealing for 1 h at 1300 °C.

FIG. 2. Atom map of a 2-nm-thick slice through the central region of a particle in MA957 annealed for 1 h at 1300 °C. Each $TiO^{2+}$ ion is represented by one sphere. The Y atoms are preferentially located in the core regions of the particle.

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