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Understanding the influence of grain size on α’ Cr precipitation in Fe-21Cr-5Al alloy during thermal aging using atom probe tomography

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FeCrAl alloys are considered as a candidate material for fuel cladding in commercial light water reactors (LWRs) replacing Zircaloy owing to their high temperature strength and corrosion resistance in steam environments (around 1000oC) [1]. However, FeCrAl alloys also suffer from embrittlement after aging at 475 oC and lower (the operating temperature of LWRs falls into this range), due to α’ Cr precipitation that results in an increase in the DBTT [2, 3]. This phenomenon is detrimental to the structural performance and also to the corrosion resistance of KD. Therefore, it is important to deeply study and understand α’ Cr precipitation in high Cr ferritic alloys. There exists no systematic study that looks into the influence of grain size on α’ Cr precipitation during isothermal aging. This study will be the first to systematically study the precipitation of nanoscale α’ Cr (5-10 nm) as a function of grain size [coarse-grained (CG, grain diameter >1 μm; ultrafine grained (UFG, 100 nm < grain diameter < 1 μm); nanocrystalline (NC, grain diameter < 100 nm)] during isothermal aging at or below 475 oC.

Kanthal-D (KD, Fe-21Cr-5Al alloy) commercial bar stock was manufactured into UFG and NC samples using two forms of severe plastic deformation (SPD), equal channel angular pressing (ECAP) and high-pressure torsion (HPT), respectively. ECAP KD had a non-homogenous microstructure with multi-modal grain size distribution revealed using electron-back scatter diffraction. Average grain size of HPT KD was estimated to be 75 ± 40 nm using bright-field transmission electron microscopy (BF-TEM). Following SPD, CG KD, ECAP KD, and HPT KD were isothermally annealed at 450 oC and 500 oC for 500 h.

Vickers microhardness was measured after aging each of the samples at 450 oC and 500 oC, the results of which are shown in Figure 1. The hardness of CG, ECAP, and HPT KD after aging at 450 oC was found to be 332.6 ± 6.0 HV, 407.5 ± 5.3 HV, and 442 ± 3.7 HV, respectively. The hardness of CG, ECAP, and HPT KD after aging at 500 oC for 500 h was found to be 379.6 ± 9.2 HV, 414 ± 3.9 HV, and 339.1 ± 3.5 HV, respectively. Unaged CG, ECAP KD, and HPT KD have a hardness of 227.8 ± 2.05 HV, 332.7 ± 10.2 HV, and 595.4 ± 8.5 HV, respectively. Comparing the unaged and the aged specimens, CG KD shows an increase in the hardness of ~105 HV followed by ECAP KD with a hardness increase of ~75 HV after aging at 450 oC. In addition, there is only an increase of ~6.5 HV in the hardness of ECAP KD after aging at 500 oC compared to ECAP KD after aging at 450 oC. However, CG KD aged at 500 oC shows an increase of ~47 HV in hardness compared to CG KD aged at 450 oC.

The increase in hardness is a consequence of α’ Cr precipitation as can be seen in Figure 2. It was estimated using cluster analysis in IVAS (Integrated Visualization and Analysis Software) that the number density and average radius of α’ Cr precipitates (ppts.) increased with increase in annealing temperature in CG KD. In ECAP KD, it was estimated that the number density and average radius of α’ Cr ppts. were comparable for both the annealing temperatures explaining the comparable microhardness after aging at 450 oC and 500 oC. It can be inferred that in the case of ECAP KD, due to enhanced diffusion and therefore faster precipitation kinetics, the precipitation at 450 oC is already near equilibrium, and an increase in temperature of 50 oC does not really change precipitation characteristics.

In the case of HPT KD, there is a drop in hardness of ~150 HV and ~260 HV after aging at 450 oC and 500 oC, respectively. It was previously determined from a thermal stability study that HPT KD is unstable at 500 oC and that grain growth takes place after 3 h of annealing. This suggests that the drop in hardness is due to grain growth leading to loss in grain boundary strengthening.

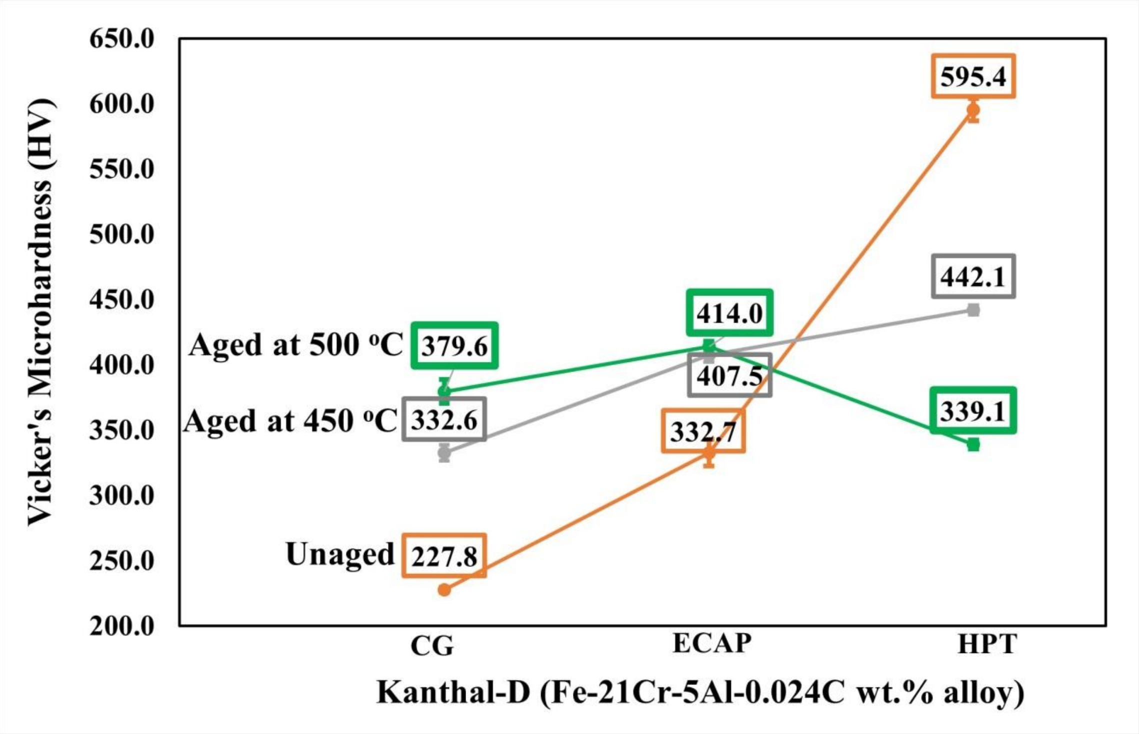

Figure 1. Figure 1. Vickers microhardness after aging of CG, ECAP and HPT KD at 450 oC, and 500 oC for 500 h.

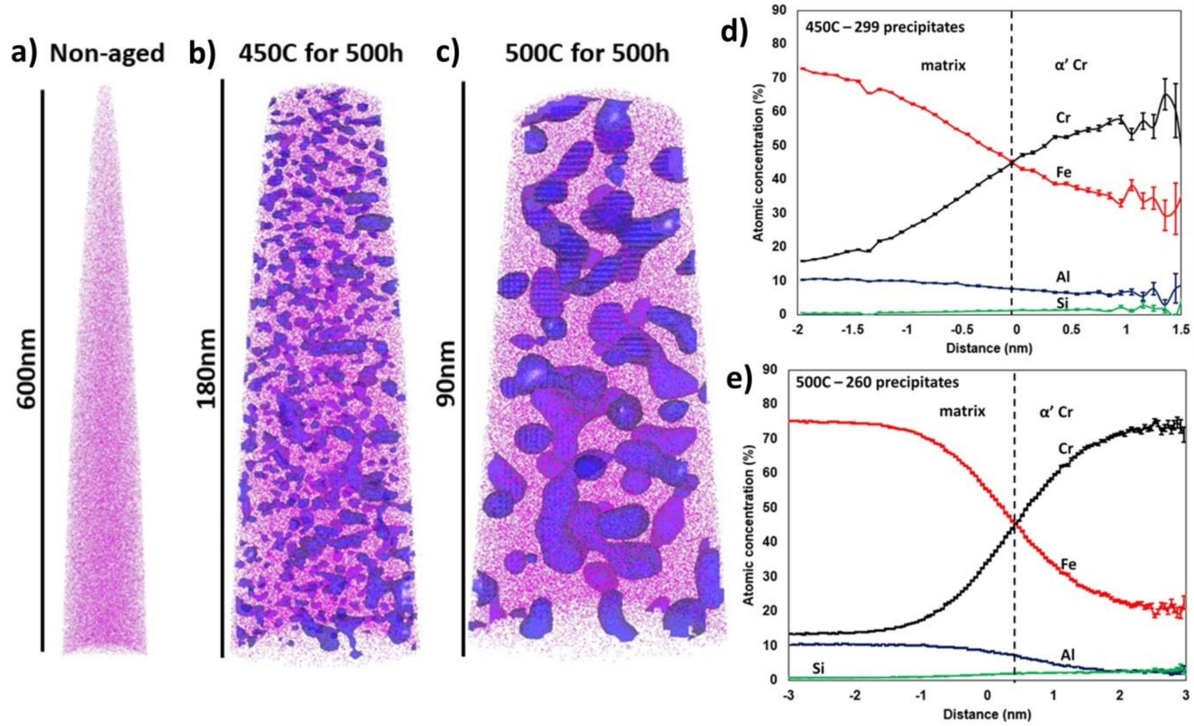

Figure 2. Figure 2. APT reconstruction of Fe atoms (pink) with 35 at.% Cr isoconcentration-surface (blue) in a) unaged, b) 450 oC, and c) 500 oC aged ECAP KD. Statistical proxigrams based on 35 at.% Cr isoconcentration-surface from d) 450 oC, and e) 500 oC aged ECAP KD.

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