Fe-Cr Alloys for Advanced Nuclear Energy Applications

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August 20, 2013

*The project objective is to exploit thermodynamic stabilization as an alternate means to control grain growth in model NFA’s at elevated temperature, with the outcome that radiation damage tolerance can be improved by increase in the interfacial area/volume ratio, while also contributing to grain size strengthening mechanisms.*
Thermodynamic Stabilization of Grain Size

The concept is that *non-equilibrium* solutes introduced by mechanical alloying can segregate to grain boundaries, producing a minimum in the excess Gibbs free energy -> metastable equilibrium of the grain size at elevated temperatures.

A binary alloy (C + stabilization solute A) regular solution mixing model developed by Trelewicz and Schuh (TS)\(^1\) properly minimizes the excess Gibbs free energy with respect to *both* interfacial solute concentration and grain boundary content – a critical step relative to previous models. The TS model includes bond interaction (chemical) enthalpy, but solute size misfit (elastic) enthalpy is ignored. In previous work, we extended the TS binary model to include elastic enthalpy, and also developed an effective computational scheme for numerical evaluation\(^2\).

A ternary alloy (CB + stabilization solute A) introduces additional chemical and elastic interactions that can be very significant. We developed a ternary model as part of the NEET program\(^3\), and use this for FeCrX alloy simulations.

Ternary model

Fe Cr A = C B A (at %)

Model Input data:

Elastic enthalpy $\Delta E < 0$

$\Delta E_{AC}$

$\Delta E_{BC}$

Chemical enthalpy $\Delta H = \omega z/4$

$\Delta H_{AC}$

$\Delta H_{BC}$

$\Delta H_{AB}$

$\Delta H > 0 \rightarrow$ phase separation

$\Delta H < 0 \rightarrow$ phase formation
Fe10Cr4Zr simulation and experimental result for 900°C

The simulation grain size at 900°C = 65 nm and the solubility limit in the bulk is 2.3 %Zr. The SAD pattern indicates the presence of fine nanoscale intermetallic precipitates.

Volume average grain size = 58 nm
Hf is stronger than Zr for retaining nanoscale grain size at higher temperatures. Choosing a stabilizer solute has several aspects in addition to the grain size range. The melting points must be high and the expected resistance to grain-boundary embrittlement should be at least neutral\(^1\). Both Zr and Hf are favorable.

Simulations for Fe$_{14}$Cr$_x$Sr and Fe$_{14}$Cr$_x$Sc

Sr and Sc are very strong stabilizers up to high temperatures. The melting points are high and the bulk solubility is low, essentially zero for Fe$_{14}$Cr$_x$Sr. Grain boundary embittance could be a concern$^1$. Preliminary experiments will be done to evaluate these as stabilizers for Fe$_{14}$Cr.

Characterization of a base ODS NFA alloy
Fe-14Cr-0.4Ti-xY$_2$O$_3$ (wt %)

All processing is done using SPEX milling

The target is $x = 0.25$. Values of $x = 10$ and 1 were used initially to investigate the $Y_2O_3$ distribution for nano and micron scale starting powders.
TEM for as milled Fe-14Cr-0.4Ti-10Y$_2$O$_3$

Results are independent of initial Y$_2$O$_3$ powder size. Grain size = 20 nm
STEM for as milled Fe-14Cr-0.4Ti-10Y₂O₃
TEM for Fe-14Cr-0.4Ti-10Y₂O₃ annealed at 1100C/1 hour

SAD (right image) isolates intermetallic precipitates
TEM for as milled Fe-14Cr-0.4Ti-1Y$_2$O$_3$

Results are independent of initial Y$_2$O$_3$ powder size. Grain size = 30 nm
TEM for Fe-14Cr-0.4Ti-1Y$_2$O$_3$ annealed at 1100C/1 hour

SAD (right image) isolates intermetallic precipitates
Microhardness vs. annealing temperature (1 hr) for Fe-14Cr-0.4Ti-0.25Y₂O₃

Hardness is measured on un-compacted powder particles.
Microstructure of the Fe-14Cr-0.4Ti-0.25Y$_2$O$_3$ after 1100°C annealing for one hour.
(a) High-angle annular dark-field (HAADF) image in STEM
(b) Bright-field image in TEM

Grain size > 100 nm
Elemental mapping of a typical oxide intermetallic precipitate (in the previous figure microstructure) by energy dispersive spectroscopy (EDS)

(a) HAADF image, (b)-(f) the major elements within the oxide are Fe, Cr, Y, Ti and O, respectively (40 nm scale bars).