Investigation of the Nanoscale (Ni,Fe)Al Precipitates in the Ferritic Superalloy by USAXS

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Introduction and Objectives

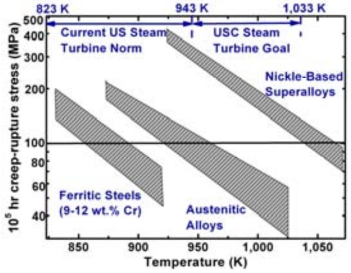


Fig. 1 10^5 hr creep rupture strength for current and potential materials for steam turbines.

●**Motivation**: to design a B2-type (Ni,Fe)Al precipitation hardened ferritic superalloy with a steady-state creep rate of 10^{-11}/s at 30 MPa and 1,033 K for ultra-supercritical steam turbines

●**Objective**: to study the effects of precipitate size and spacing on the creep behavior by ex-situ ultra-small-angle X-ray scattering (USAXS) measurements

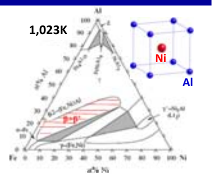


Fig. 2 Fe-Ni-Al phase diagram.

Why USAXS at APS?
- Probe the bulk
- Resolve large-dimension structure
- Tune to high energy

Experimental

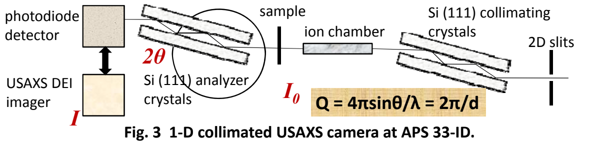


Fig. 3 1-D collimated USAXS camera at APS 33-ID.

Measured Q range: 10^{-4} ~ 0.5 Å^{-1}
Beam energy: 16.85 keV
Beam size: 1.6 x 0.8 (mm)
Foil thickness: ~ 50 µm
Data reduction and desmearing:
Indra and Irena marcos for Igor Pro

Table 1 Alloy composition (in wt.%)

Alloys #	Fe	Al	Cr	Ni	Mo	Zr	B
FBB-3	66.3	10	10	10	3.4	0.25	0.005
FBB-7	68.3	8	10	10	3.4	0.25	0.005
FBB-8	69.8	6.5	10	10	3.4	0.25	0.005
FBB-17	70.3	6	10	10	3.4	0.25	0.005
FBB-9	71.3	5	10	10	3.4	0.25	0.005
FBB-12	72.3	4	10	10	3.4	0.25	0.005

Materials:
- Drop cast, homogenized at 1,473 K for 30 min, and aged at 973 K for 100 hrs
- Compressive crept at 140 MPa and 973 K for 100 hrs with size of 6 x ϕ 4 (mm)

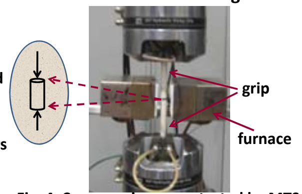


Fig. 4 Compressive creep tested by MTS.

Theoretical Model

Absolute intensity distribution *-- Fourier transformation of microstructure*

Number of density Of precipitates; Difference in scattering-length density between two phases; Scattering from grain boundaries (Porod's Law); Incoherent scattering intensity (background)

One Population
$$I(Q) = n_p \,|\Delta\rho|^2\, V_p^2 P(Q) S(Q) + AQ^{-b} + I_{INC}$$

Form factor – particle size and shape

$$P(Q) = \left(\frac{4\pi}{3}\right)^2 \int F^2(Q,x) * N(x,R,\delta) * R^6 dx$$

Sphere $F(Q,x) = [\sin(Qx) - Qx\cos(Qx)]/(Qx)^3$

Gaussian Law - polydispersity

$$N(x,R,\delta) = \exp\left[-(x-R)^2/2\delta^2\right]/\sqrt{2\pi\delta^2}$$

Structure factor – inter-particle correlation

$$S(Q,L,\sigma) = 2\left\{\frac{1-\exp\left(-\frac{Q^2\sigma^2}{4}\right)\cos(QL)}{1-2\exp\left(-\frac{Q^2\sigma^2}{4}\right)\cos(QL)+\exp\left(-\frac{Q^2\sigma^2}{2}\right)}\right\}-1$$

Non-linear least square fitting for 8 parameters: $n_p\,|\Delta\rho|^2\, V_p^2, I_{INC}, A, R, \delta, L, \sigma, b$

Validate the fitted parameters by TEM precipitate characterizations

Results and Discussions

● The model shows agreement with TEM results.

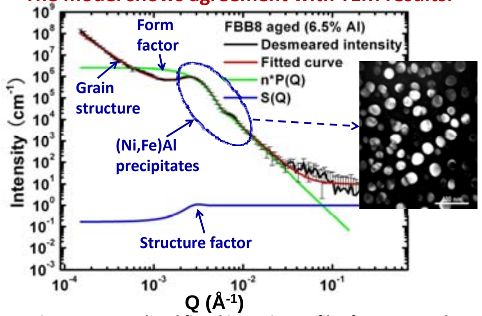


Fig. 5 Measured and fitted intensity profiles for FBB8 aged alloy, compared with dark-field TEM micrographs.

Table 2 Precipitate parameters for FBB8 aged alloy.

Parameters (unit: Å)	USAXS	TEM
Radius of precipitates (R)	637.29 ± 22.54	650
Variance of R (δ)	156.84 ± 17.80	125
Inter-particle spacing (L)	1761.71 ± 49.17	1820
Variance of L (σ)	1018.74 ± 36.95	940

● After creep deformation, inter-particle spacing increases and variance of spacing decreases.

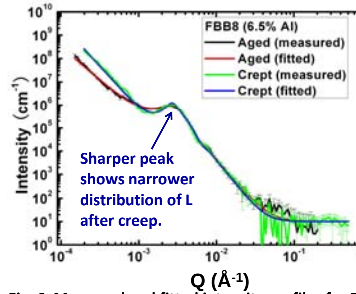


Fig. 6 Measured and fitted intensity profiles for FBB8 aged and creep-deformed alloy.

Table 3 Fitted parameters for FBB8 aged and deformed alloys.

Parameters (unit: Å)	Aged	Crept
Radius of the precipitates (R)	637.29 ± 22.54	595.15 ± 8.49
Variance of R (δ)	156.84 ± 17.80	146.39 ± 6.75
Inter-particle spacing (L)	1761.71 ± 49.17	2109.56 ± 14.10
Variance of L (σ)	1018.74 ± 36.95	847.06 ± 9.49

● With Al% increases, both precipitate size and spacing decrease. Steady-state creep rate is reduced by one order of magnitude, probably because of enhanced resistance of particles to dislocation motion.

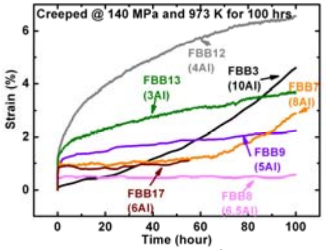


Fig. 7 Typical creep curves for creeped alloys at 140 MPa and 973 K for 100 hours.

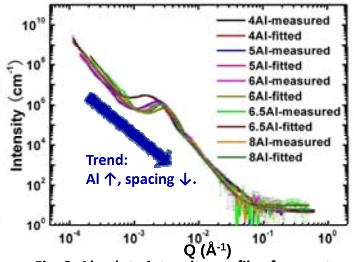
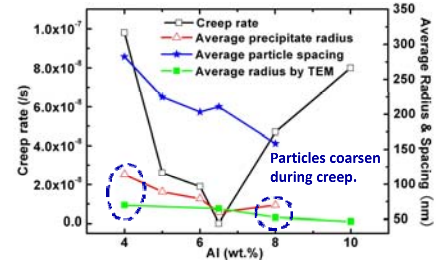


Fig. 8 Absolute intensity profiles for crept alloys with varying Al concentrations.



- Inter-particle spacing is dominant on creep rate.
- Correlation is not valid for FBB7 (8% Al), which will be further studied.
- Coarsening during creep deformation is evident in FBB7 (8% Al)and FBB12 (4% Al).

Fig. 9 Creep rate, particle radius, and spacing as a function of Al%.

● Dislocation-particle interactions (threshold stress behavior)

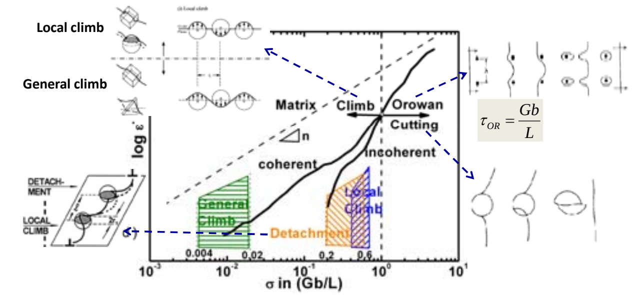


Fig. 10 Schematic creep behavior of particle strengthened materials. The stress is given in units of the classical Orowan stress.

Summary

●A theoretical model that incorporates polydisperse form factor and a phenomenological structure factor is employed to predict the precipitate parameters in the ferritic superalloy. Fitted results compare favorably with complementary TEM characterizations.

●As Al% increases, average inter-particle spacing becomes smaller, associated with an improvement in creep resistance due to inhibited dislocation motion by precipitates. After creep, particle coarsening is observed.

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