Evolution under Irradiation of Optimized Austenitic Steel For Gen-IV Reactors. Impact on Fuel Cladding Properties and Performances.

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An important class of swelling-resistant alloys used in fast-reactor are the cold-worked, titanium-stabilized austenitic stainless steels (15-15Ti AIM1 in France, DIN1.4970 in Germany, optimized D9 in the US and AFIC1 in India). An advanced 15-15Ti alloy was selected for the fuel cladding of the initial core of the fast-reactor French project ASTRID [1]. To extend its qualification at high dose, post-irradiation examinations were recently performed on mixed-oxide fuel pins, irradiated in the Phénix fast-reactor up to 130 dpa and retrieved after Phénix shutdown. As shown in Fig. 1, destructive examinations in hot labs (CEA/LECA-STAR and CEA/LECI facilities) were done to quantify the contribution of irradiation creep and swelling to the total cladding deformation. The results are compared with the past examinations on the same material at lower dose 80 and 110 dpa [2] and confirm the very good swelling resistance of this optimized alloy at 130 dpa.

Fig. 1: a) Diametral strain of an irradiated 15-15Ti (optimized) cladding at a maximum dose of 130 dpa. b) Maximum diametral strain and swelling versus the maximum cladding dose.

The evolution of 15-15Ti microstructure under neutron irradiation was investigated by TEM [3]. As shown in Fig. 2, the irradiated microstructure features complex interactions between vacancy clusters, precipitates (fine titanium-carbide and coarser Cr-rich precipitates, G phase) and dislocations network. The simulation of irradiation effects on advanced 15-15Ti using ions irradiation in the JANNUS-Saclay facility will be presented. The comparison between single (2 MeV Fe²⁺) and dual beams (2 MeV Fe²⁺ - 1 MeV He⁺) experiments stresses the importance of helium in swelling mechanisms. As in neutron irradiation, heterogeneous nucleation of vacancy clusters is evidenced but marked differences in precipitation mechanisms are observed between neutron and ions irradiation.

Fig. 2: TEM micrographs of optimized 15-15Ti samples showing cavities microstructure and the interaction between precipitates and voids : a) neutron irradiation 88 dpaNRT 470°C, b) ions irradiation 90 dpaKP 630°C.

The modeling of irradiation evolution in such complex alloy is challenging. The presentation will summarize the current efforts to develop a model capable of predicting temperature, dose, and flux effects on swelling using cluster-dynamics techniques. The modeling of helium effects is also discussed. One of the future areas of work is to include the effects of precipitation (interface effects, helium and point-defects trapping, solid-solution decay etc.).

The Impact of irradiation on tensile and creep properties of 15-15Ti is also briefly discussed with special emphasis on correlations between swelling and embrittlement. At high temperature >600°C, limited data on cold-worked 316Ti and 15-15Ti shows that creep rupture time and ductility are reduced and intergranular failure is enhanced.

References

- [1] Le Flem et al. "Status of the French R&D on materials for the core components of ASTRID", International Congress on Advances in Nuclear Power Plants, ICAPP 2014, Charlotte, USA, April 6-9 2014.
- [2] A. Maillard et al. "Swelling and Irradiation Creep of Neutron Irradiated 316Ti and 15-15Ti steels", Effects of Radiation in Metals : 16th International Symposium, ASTM STP 1175, 1992.
- [3] C. Bisor et al., Personal communication CEA, 2012.
- [4] M. Kountchou et al. "Ions Irradiations of Cladding Materials for the Fourth-Generation Nuclear Reactors", Material Science and Technology, MS&T 2015, Columbus, USA, October 4-8 2015.

DE LA RECHERCHE À L'INDUSTRIE

EVOLUTION UNDER IRRADIATION OF OPTIMIZED AUSTENITIC STEEL FOR GENIV REACTOR.

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- Neutron irradiation and Post-irradiation examinations
- Ion irradiation
- Modeling
- Impact of irradiation on mechanical properties

MATERIALS 15-15TI

- Standard : CW 15-15Ti : used in standard French SFR Phenix assembly

Lot	C	Mn	Si	Cr	Ni	Mo	P	Ti
Standard 15-15Ti	0.08	1.0	0.3	14	14	1.3	<0.01	0.3
	0.10	2.0	0.5	16	16	1.7		0.5
15-15Ti + Si	0.08	1.0	0.7	14	14	1.3	<0.01	0.3
	0.10	2.0	0.9	16	16	1.7		0.5
15-15Ti+Si+P	0.08	1.0	0.7	14	14	1.3	0.03	0.3
	0.10	2.0	0.9	16	16	1.7	0.05	0.5

- CW 15-15Ti + Si : tested in SFR up to 130 dpa
- CW 15-15Ti + Si + P : **AIM1**, irradiated in Phénix up to 90 dpa post-irradiation examinations in progress

Fully austenitic, grain size $\sim 30 \mu\text{m}$

Thermal and mechanical twins,
strongly strained grain

TiN and TiMoC primary carbides

No obvious texture

Austenite

Fig. 9. Three types of annealing twin morphologies observed in f.c.c. metals and alloys. A is a grain corner twin; B is a complete parallel-sided twin; C is an incomplete parallel-sided twin.

CW 15-15Ti (+Si+P)

NEUTRON IRRADIATION

NEUTRON IRRADIATION IN PHENIX

MINOS

- A long process !
- Cladding tube 15-15Ti+Si fabricated in 1981
- First irradiation from 1982 to 1985 up to 80 dpa
- Re-irradiation from 1986 to 1987 up to 110 dpa
- Third Re-irradiation up to 130 dpa from 1994 to 1998
- Assembly discharged in 2001
- PIE started in 2012

- CEA-Cadarache (LECA hot-lab facility)
- Metrology, γ spectrometry
- Metallography

- Cutting (CEA-Cadarache – DEC - LECA – O. Descombin et al.)

- Chemical dissolution HNO_3 to remove fuel (CEA-Marcoule – E. Buravand et al.)

- Density measurements by immersion at CEA-Saclay-LECI (M. Sonnaert, P. Bottin et al.)

NON-DESTRUCTIVE AND DESTRUCTIVE

- Non destructive : diameter profile $\rightarrow$ total strain
- Destructive : Density by immersion $\rightarrow$ swelling

Irradiation creep by
Fission-gaz pressure

Irradiation
Swelling = $\Delta V/3V$

← Fuel Column →

COMPARISON WITH PREVIOUS DATA

TEM OBSERVATION

Standard 15-15Ti (sample)
88 dpa – 467°C

G phase ($\text{M}_6\text{Ni}_x\text{Si}_y$) / M_6C
attached to cavities

DISLOCATION LOOPS.

ION IRRADIATION

PRINCIPLE

One side electropolishing

SINGLE BEAM : Fe^{2+} (1MEV) $T=630^\circ\text{C}$

M. Kountchou et al.

CAVITIES : DUAL BEAM Fe^{2+} - He^{2+} ($T=630^\circ\text{C}$)

CAVITIES : DUAL BEAM Fe^{2+} - He^{2+}

15-15Ti (Si+P) ($\text{Fe}^{2+} + \text{He}^+$), 45 dpaKP

15-15Ti (Si+P) ($\text{Fe}^{2+} + \text{He}^+$), 90 dpaKP

DISLOCATION LOOPS

$B \sim [110]$ et $g = (1-13)$

- Still here !

MODELING

CRESCENDO Framework (T. Jourdan et al.CEA/EDF)

$$\frac{dC_n}{dt} = \sum_{m \in \mathcal{M}} J_{n-m,n} - \sum_{m \in \mathcal{M}} J_{n,n+m}$$

m : mobile species

15-15Ti (+Si +P)
Annealed

B Rouxel et al.

Submitted to JNM

IVEM (ANL)

1Mev Kr²⁺

600°C

Radiative recovery

Thermal recovery

Frank Loop
unfaulting

**Dislocation
Network
evolution**

a lattice parameter

V_{at} atomic volume

$c_{bh,1}$ first constant for Bardeen Herring sources

$c_{bh,2}$ second constant for Bardeen Herring sources

$c_{a,1}$ first constant for annihilation of dipoles

$c_{a,2}$ second constant for annihilation of dipoles

σ standard deviation of the dislocation distribution

$E_{2i,i}^b$ binding energy of di-interstitials

$E_{2v,v}^b$ binding energy of di-vacancies

E_i^f formation energy of interstitials

E_v^f formation energy of vacancies

μ shear modulus

ν Poisson's ratio

γ stacking fault energy

$b = b_{\pm}$ Burgers vector for Frank loops

Z_i^l absorption efficiency of Frank loops for interstitials

Z_v^l absorption efficiency of Frank loops for vacancies

Z_i^d absorption efficiency of dislocations for interstitials

Z_v^d absorption efficiency of dislocations for vacancies

E_i^m migration energy of interstitials

E_v^m migration energy of vacancies

$D_{0,i}$ diffusion prefactor for interstitials

$D_{0,v}$ diffusion prefactor for vacancies

ΔV_i relaxation volume of interstitials

ΔV_v relaxation volume of vacancies

ρ initial dislocation density

Dislocation evolution

Binding energy for
emission rate

Bias, absorption efficiency

Diffusion coefficients of
defects

ELECTRON IRRADIATION

Exp Data from Dubuisson et al.

- Standard 15-15Ti
- Annealed 1150°C – 10mn
- 475 and 500°C
- $\sim 3.5 \cdot 10^{-3}$ dpa/s
- Beam direction $\langle 110 \rangle$
- Foil thickness (~ 300 nm)

CLUSTER DISTRIBUTION

FLUX – DPA RATE EFFECT

Temperature Shift

EFFECT OF COLD WORKING

Incubation dose !

IMPACT ON MECHANICAL PROPERTIES

- Axial tensile tests (in hot cells)

Softening at high temperature

CW 15-15Ti (+Si)

Hardening below 500°C

Reduced ductility

MINOS

- Axial tensile tests in hot cells
- CW 15-15Ti (low & high Si, low P)

CONCLUSION

- Neutrons are the only way to qualify a real (complex) material in irradiation
- Ions/Electrons are the shortest/cheapest way to perform R&D (mechanisms understanding, material comparison)
- Model are essential to rationalize experimental data (dose, temperature, dose rate, helium effect)
- But the road towards a mechanistic (multiscale) and predictive model of real alloy like 15-15Ti is still long

CONCLUSION : MANY PEOPLE INVOLVED !

- M. Kountchou, B. Rouxel, L Mateus-Freire (Phd **CEA-Saclay**)
- JL Seran, M. LeFlem, P. Gavaille, C. Bisor, M. Jublot, E. Piozin, P. Bottin (**CEA-Saclay** DEN/DANS/DMN)
- O. Descombin, T. Martella, L. Loubet (**CEA-Cadarache** DEN/DANS/DEC-LECA)
- M. Masson (**CEA-Marcoule**, Phénix)
- E. Buravand (**CEA-Marcoule**, Atalante)
- Jannus Team : (**CEA-Saclay** SRMP & SRMA)