Historical Perspective - ODS alloy Development

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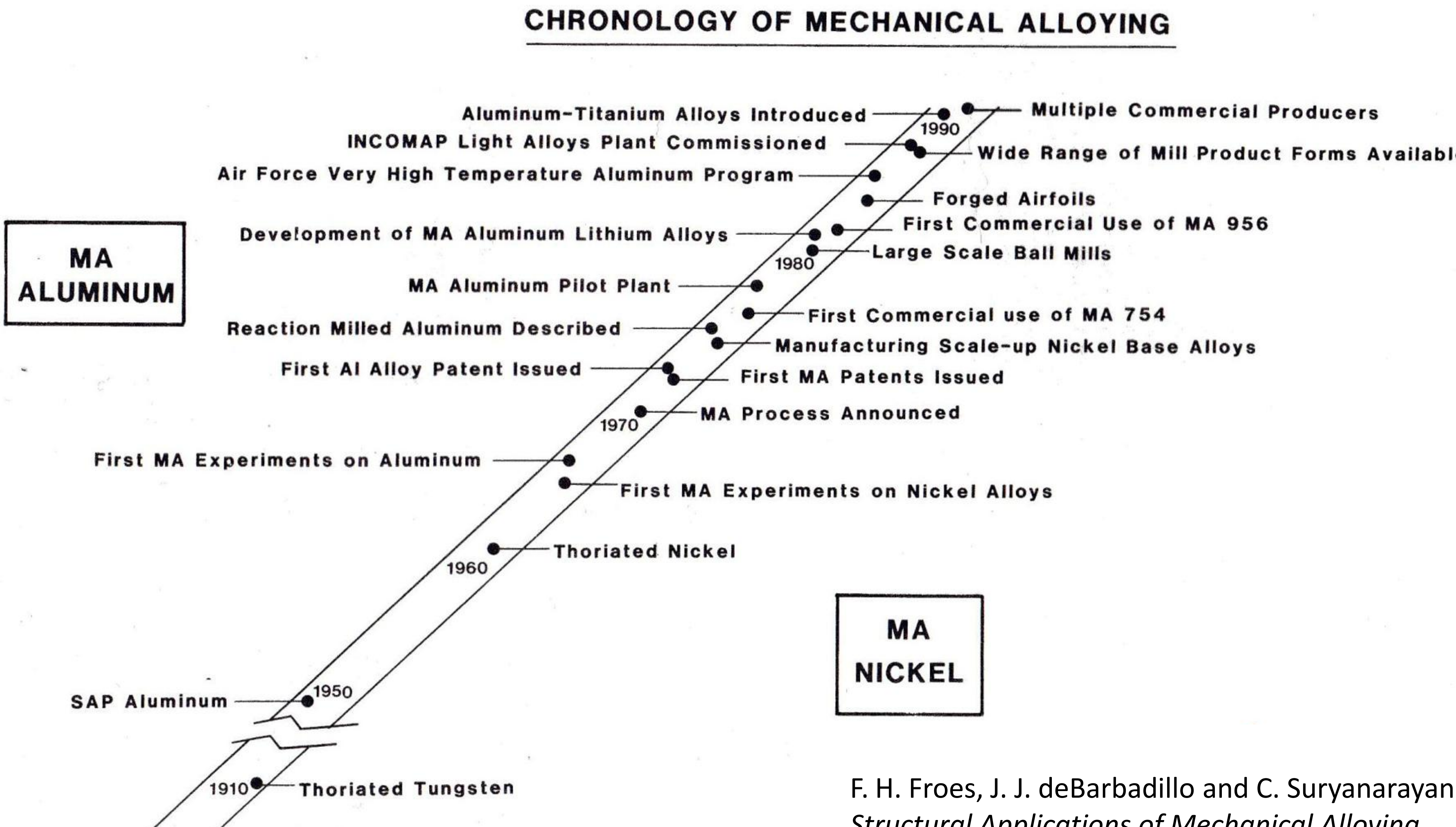
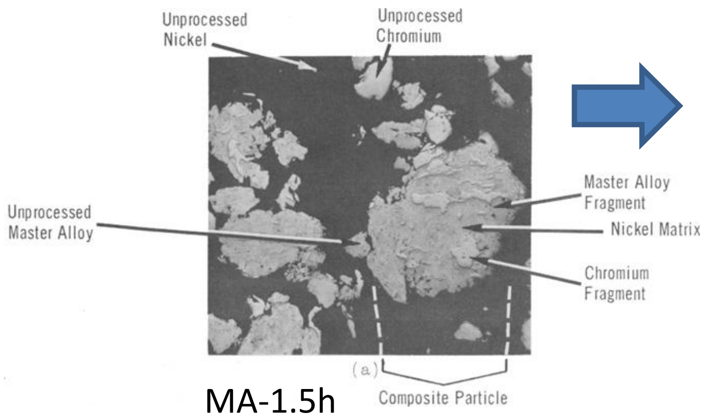
Mechanical Alloying (MA): Origins

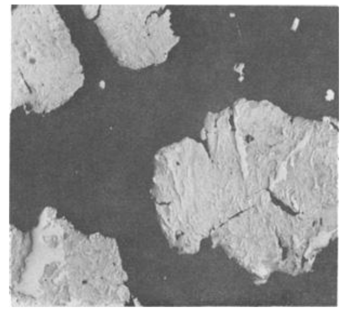


F. H. Froes, J. J. deBarbadillo and C. Suryanarayana
Structural Applications of Mechanical Alloying
Eds. Froes and deBarbadillo, ASM Intl., 1, 1990

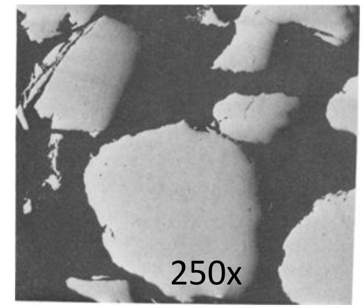
MA: Yttriated – NiCrAlTi superalloy



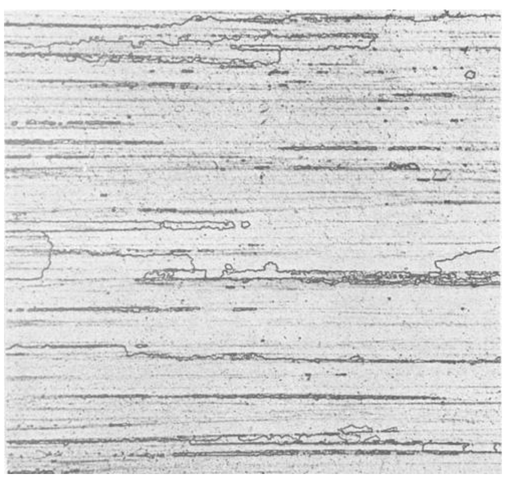
MA-1.5h

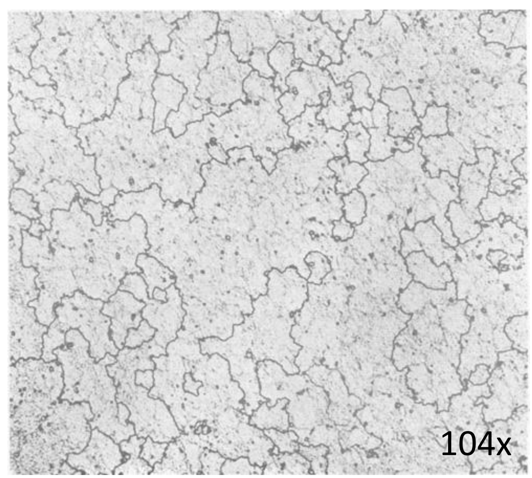


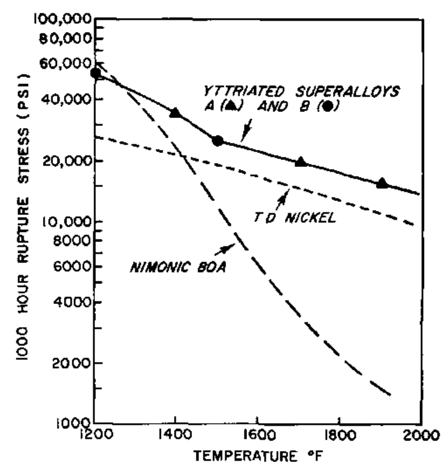
MA-9.5h



MA- 40h

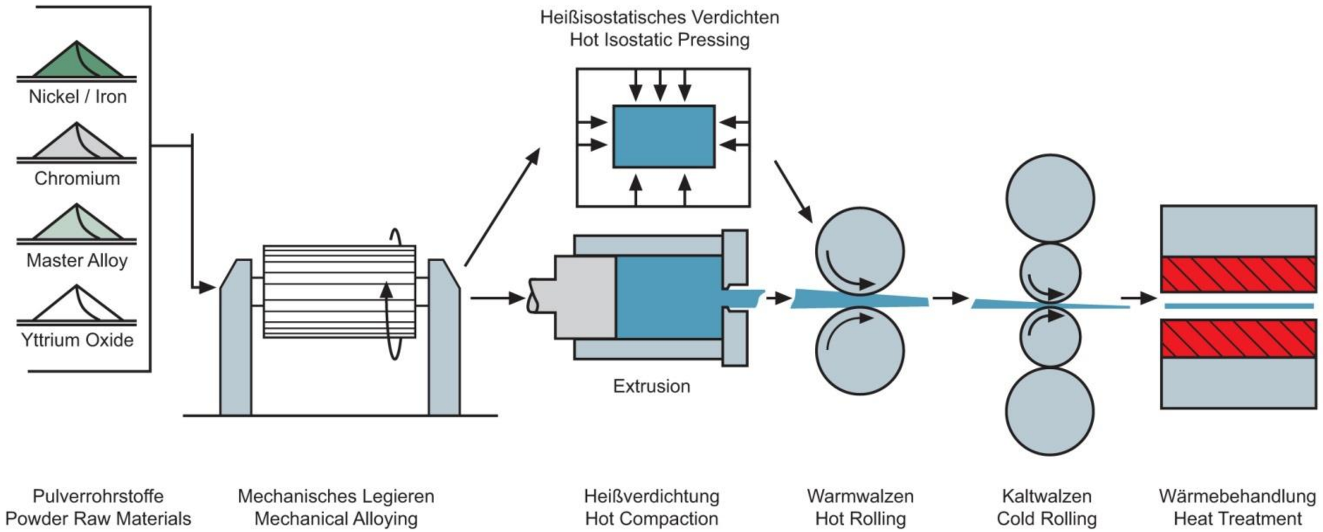


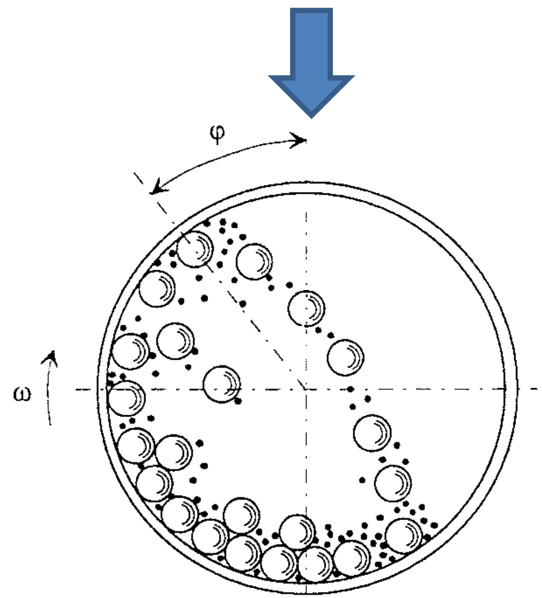




(John S. Benjamin Met Trans **1**, 1970, 2943)

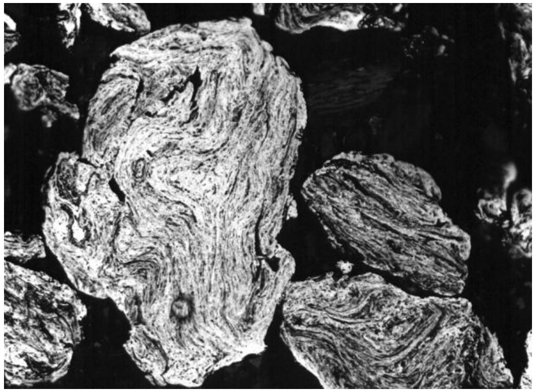
MA: the process



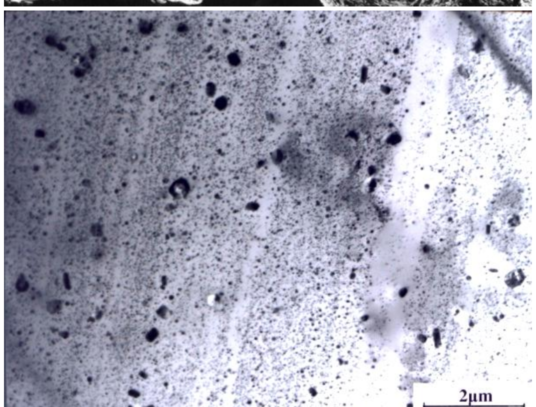


Ball Mill	Capacity	Milling duration
Shaker	g	hours
Planetary	↓	↓
Attritor		
Horizontal	100's kg	Days

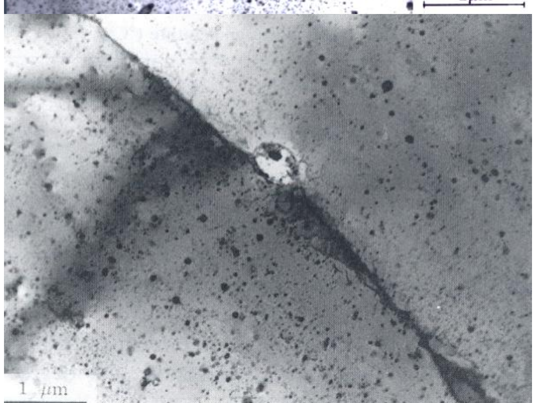
MA: ODS FeCrAl microstructures



ODM751



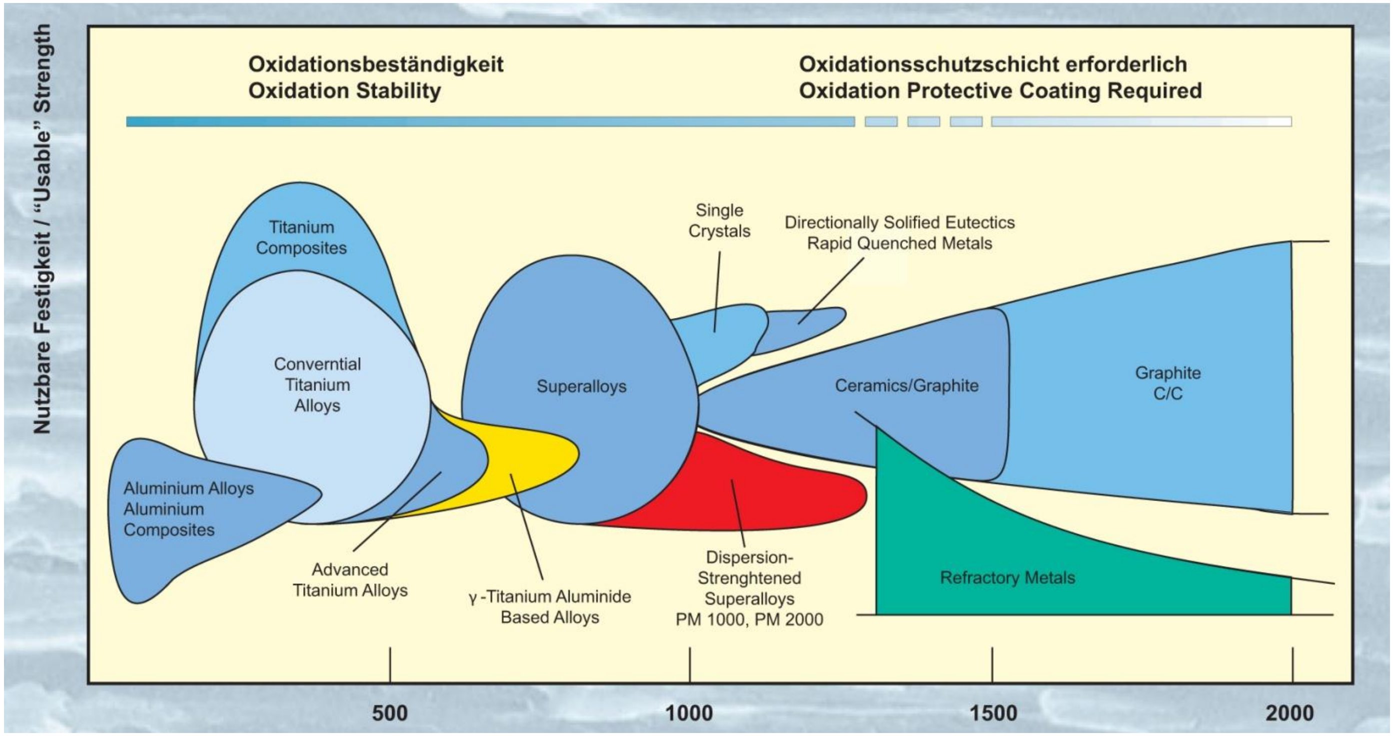
PM2000



MA956

PM2000 bar (13mm diam. , grain class 6)

ODS alloy (FeCrAl) performance



Properties

Creep resistance at up to $0.85T_m$

Oxidation resistance

Issues

Joining (fusion techniques)

Creep anisotropy

Cost

Semi-finished product size	Price (kg)
Large diameter bar	$50
Thin foil	$275

Previous commercial ODS alloys

Alloy	Composition (wt %)									
	Fe	Ni	Cr	Al	Ti	Mo	W	C	Y_2O_3	Other
MA956	bal.	-	20	4.5	0.5	-	-	0.05	0.5	
MA754	1.0	bal.	20	0.3	0.5	-	-	0.05	0.6	
MA758	-	bal.	30	0.3	-	-	0.5	0.05	0.6	
MA760	1.2	bal.	19.5	6.0	-	-	3.4	0.06	1.0	
MA6000	-	bal.	15	4.5	2.5	2.0	4.0	0.05	1.1	2.0Ta; 0.15Zr
MA957	bal.	-	14	-	0.9	0.3	-	0.01	0.25	
PM2000	bal	-	19	5.5	0.5	-	-	0.05	0.5	
PM1000	3.0	bal.	20	0.3	0.5	-	-	0.05	0.6	
PM3030	-	bal.	17	6.0	-	2.0	3.5	0.05	0.9	2.0Ta; 0.15Zr
ODM751	bal.	-	16.5	4.5	0.6	1.5	-	0.05	0.5	

Special Metals Corporation (MA alloys)
Plansee GmbH (PM alloys)
Dour Metal S.A. (now Dour Metal s.r.o.) (ODM751)

ODS Alloy Applications

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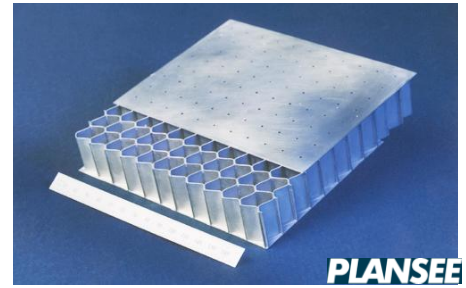
Industry sector	Alloy base	Component/application
Aerospace / military	Fe	Gas turbine combustor liners
		Fuel nozzle shrouds
	Ni	Turbine, compressor blades, **nozzle guide vanes**
	Al	Low density aerospace forgings
		Spars, ribs, wing tip panels, compressor vanes
		Torpedo hulls
Automotive	Fe	**Diesel fuel inlet atomiser**
		Turbocharger scrolls
	Ni	Recombustors
	Al	Composite pistons, compressor rotors, vanes, impellors
Power generation	Fe	**Burner nozzles,** swirlers
		High temperature heat exchangers
	Ni	Gas turbine compressor blades
Furnace furniture etc	Fe	**Nozzles, stirrers, gobbers, insert tubes** – glass
		Furnace skid rails, **charge carriers**
		Creep/fatigue rig test bars
		Heating element wires

ODS Alloy Suppliers

Date	Company	Location
1980	INCO Alloys	USA/UK
1990	INCO Alloys	USA/UK
	Alcoa	USA
	Dow Chemicals	USA
	Howmett	USA
	Exxon/Raufoss	USA/Norway
	Dour Metal SA	Belgium
	Krebsoge	Germany
	Siemens AG	Germany
	Plansee GmbH	Germany
	Kobe Steel	Japan
	Sumitomo Metal Ind.	Japan
	NRIM	Japan
	Showa	Japan

ODS alloy components

PM2000 weldless preoxidized burner tubes (diam.77mm) - for continuous annealing lines (Steel industry)



PM1000 Brazed honeycomb component (courtesy Allison Engine Company)

Low NO_x burner nozzles made of PM2000

PM1000 guide vanes

Charge carrier in PM2000

Alloys produced by MA – mid 1990’s

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Class of Material	Alloys
Light alloys	Al-Mg-Li
ODS alloys	FeCrAl + yttria; Al-Fe-Ce (RS)
MMCs	Al-SiC
Intermetallics	Cr_3Si; $MoSi_2$; NiTiAl
Cermets	Ni-TiC (reactive milling)
Metastable systems	Fe-Zr; Co-Zr (amorphous/glassy)
Nano-crystalline materials	Cu-Ta; Fe-Ta-W
Superconductors	Si-Pb (metallic); Ba-Ln-Cu (ceramic)
Super-corroding alloys	Mg-Fe
Magnetic materials	Fe-Nd-B

ODS Ferritics for nuclear applications

SCK•CEN
STUDIECENTRUM VOOR KERNENERGIE
CENTRE D'ETUDE DE L'ENERGIE NUCLEAIRE

J J Huet et al.
~1975 - 1985

Development of **creep resistant ODS ferritic tubes** for (liquid Na cooled) fast reactor fuel pins

Ferritic	Swelling resistance (neutrons)
ODS	Improved creep resistance (500-700^{0}C) He traps (particle/matrix interfaces)

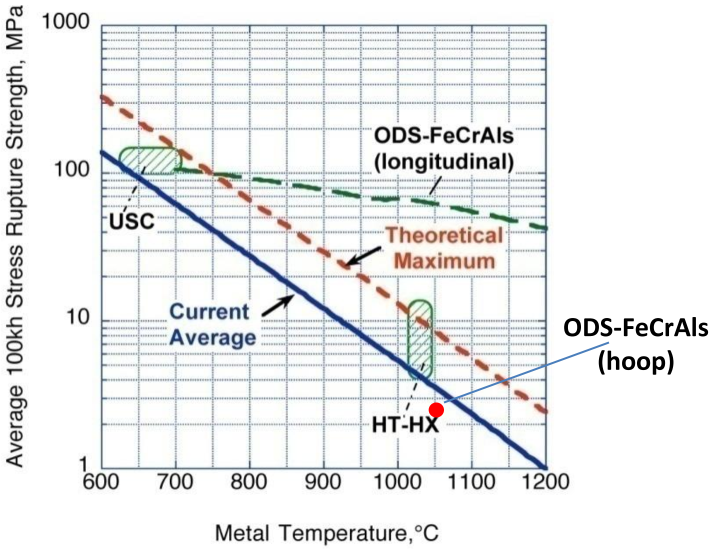
composition;
tensile/creep;
tube fabrication/control;
corrosion(Na);
irradiation behaviour;
acid dissolution

Composition (wt %)

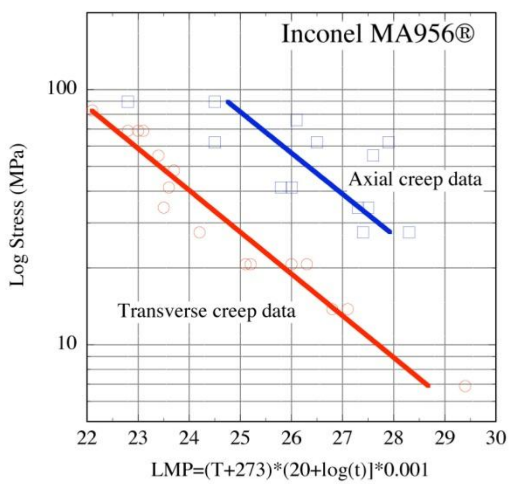
Alloy	Fe	Cr	Al	Ti	Mo	W	C	Y_2O_3	Ti_2O_3
DT2906	bal.	13	-	2.9	1.5	-	0.05	-	1.8
DT2203Y05	bal.	13	-	2.2	1.5	-	0.05	0.5	-
DT	bal.	13	-	2.9	1.5	-	0.05	-	1.8
DY	bal.	13	-	2.2	-	-	0.05	0.5	0.9
MA957	bal.	14	-	1.0	0.3	-	0.01	0.25	-

CEN/SCK Mol (DT2906, DT2203Y05) | Dour Metal SA (DT, DY) | Inco (MA957)

ODS Ferritics for HT Heat Exchangers



Actual vs Theoretical Creep-Rupture Strength: Wrought /Cast Ni-Fe-Cr Alloys*



1990 - 2000+

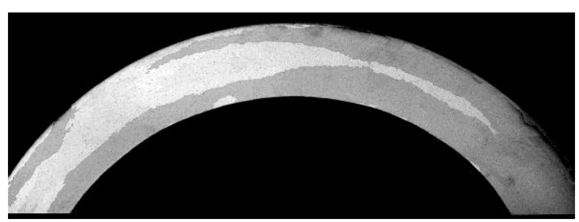
Electricity generation → higher temperatures

Increase efficiency of energy conversion

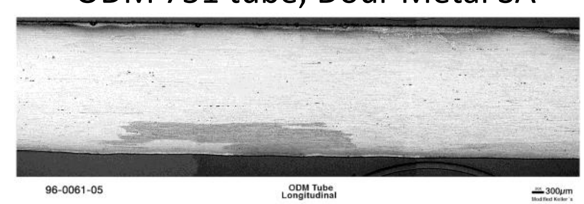
Advanced closed-cycle gas turbine systems: Natural gas fired
Coal/biomass offset

High Temperature Heat Exchanger
1100^{0}C + metal T^{0}C
ODS FeCrAl **tubed**

- British Gas ODS alloy HTHE Demonstrator plant (UKDTI funding)
- High Temperature Advanced Furnace (HITAF)
  High Performance Power Systems (HIPPS):
  United Technologies Research Center (UTRC) et al
  USDOE funding

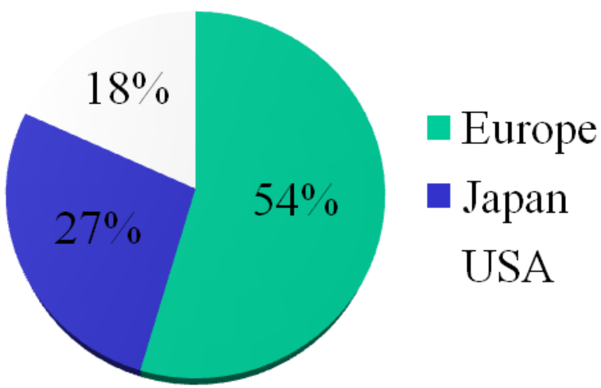


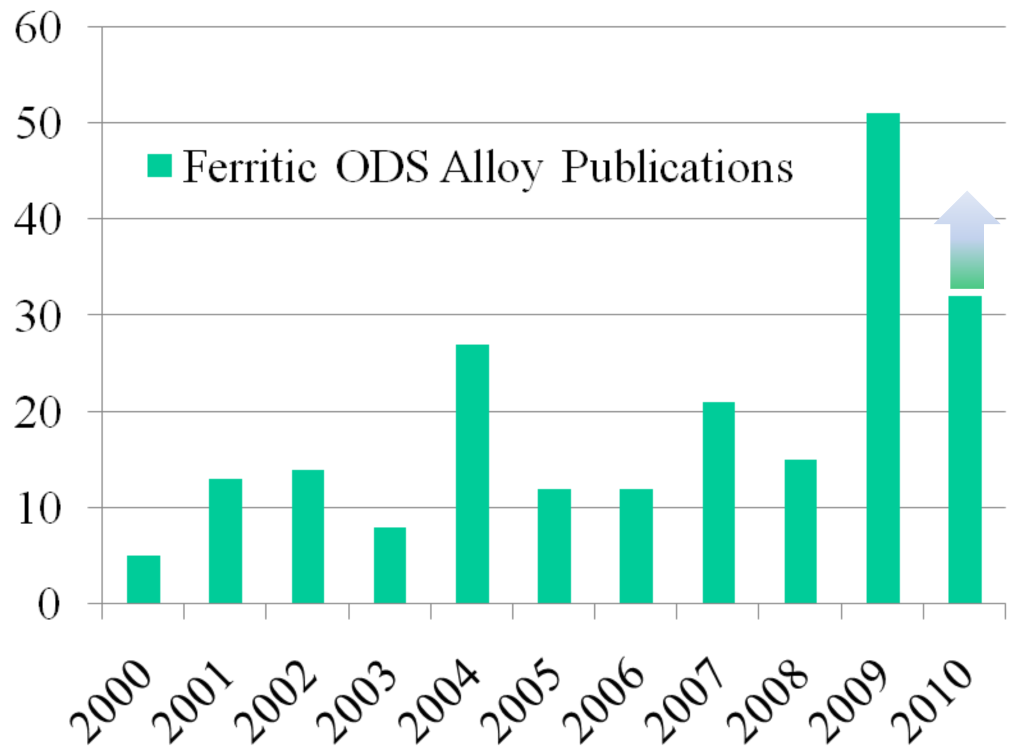
ODM 751 tube, Dour Metal SA



* I.G. Wright, B.A. Pint, and Z.P. Lu, *'Overview of ODS alloy development,' 19th Annual* Conference on Fossil Energy Materials, Knoxville, Tennessee, May 9th-11th, 2005.

ODS Ferritics: publications 2000-10

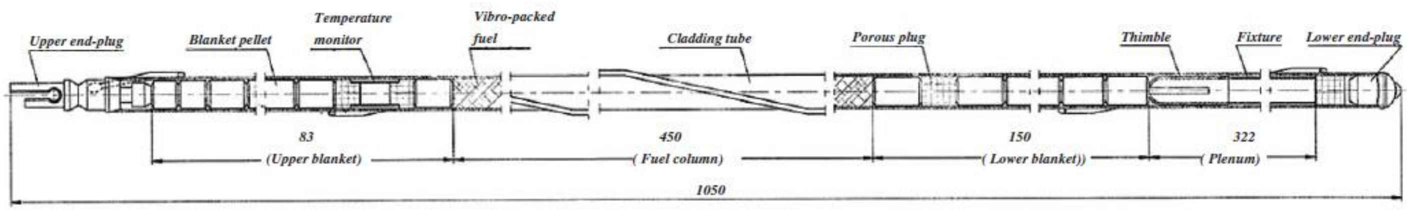


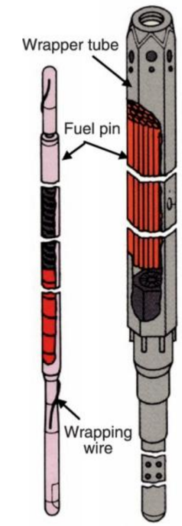


85%+ ODS FeCr publications from nuclear sector

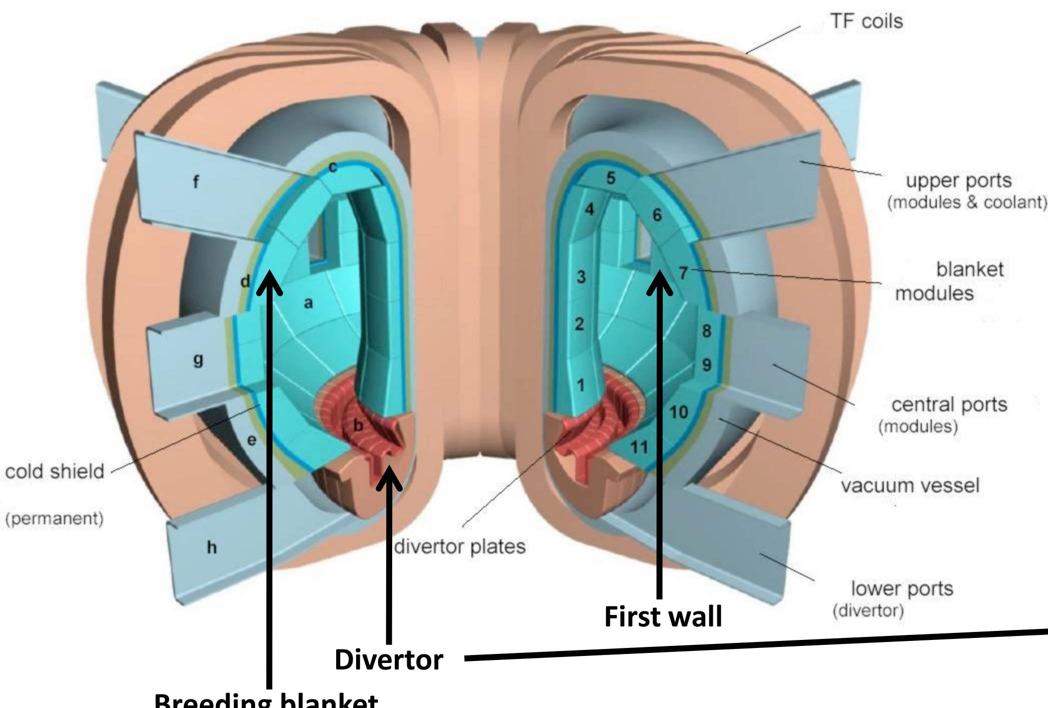
ODS Ferritics: nuclear applications

Fast Reactor fuel cladding (tubing)

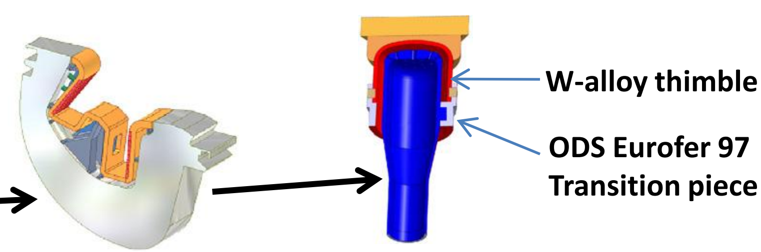




ODS fuel pin structure: BOR-60 irradiation test, proto-type FBR MONJU, Japan



Fusion reactor schematic



ODS Ferritics: nuclear applications

Power Reactor	Components	Irradiation conditions	Operating Temperature
Fast Reactor (~1MeV neutrons)	Fuel element	~ 200 dpa ~ 0.1appm He/dpa ~ 0.1appm H/dpa	~400 to ~700 ^{0}C (Na-cooled)
DEMOnstration **Fusion Reactor** (14.1 MeV neutrons)	First Wall Tritium Breeding blanket Blanket Divertor	~50 dpa + ~12 apppm He/dpa ~45 appm H/dpa	~320 to ~700 ^{0}C

Alloy design for Reduced Activation	
Low activation elements	Fe, Cr, V, Ti, W, Ta, Si, C
Long-lived radionuclides	Mo, Ni, Nb, Al, N

Alloy design issues

High temperatures
High stresses (time varying)
Aggressive environment
Intense neutron irradiation

Dimensional stability
Thermal/irradiation creep
Void swelling (α/α')

Mechanical properties
Strength, ductility
fracture toughness
Creep rupture (ODS)
Creep-fatigue

Resistance to radiation damage
Irradiation hardening/embrittlement
He embrittlement (Y-Ti-O, NF)
Radiation induced segregation
Radiation enhanced diffusion

Chemical compatibility
Structural material + fuel
Structural material + coolant

Other
Workability, joining, cost etc

ODS Ferritics: nuclear applications

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Evolution of ODS Ferritic alloy compositions for nuclear applications

Alloy	Composition (wt %)									
	Fe	Cr	W	Ti	Mo	Mn	V	Ta	C	Y_2O_3	Other
ODS Eurofer 97	bal.	9	1.1	-	-	0.4	0.2	0.12	0.11	0.3	0.03N
Mm13[1]	bal.	8.9	2	0.21	-	-	-	-	0.13	0.36	0.01N; 0.05Si
12YWT	bal.	12	3	0.4	-	-	-	-	-	0.25	
14YWT	bal.	14	3	0.4	-	-	-	-	-	0.3	
MA957	bal.	14	-	0.9	0.3	-	-	-	0.01	0.25	
MA ODS-H[2]	Bal.	15	2	-	-	-	-	-	0.03	0.35	4Al; 0.8Zr
Fe–18Cr1W[3]	bal.	18.1	0.95	0.26		0.31			0.03	0.56	Si 0.3; Ni 0.2

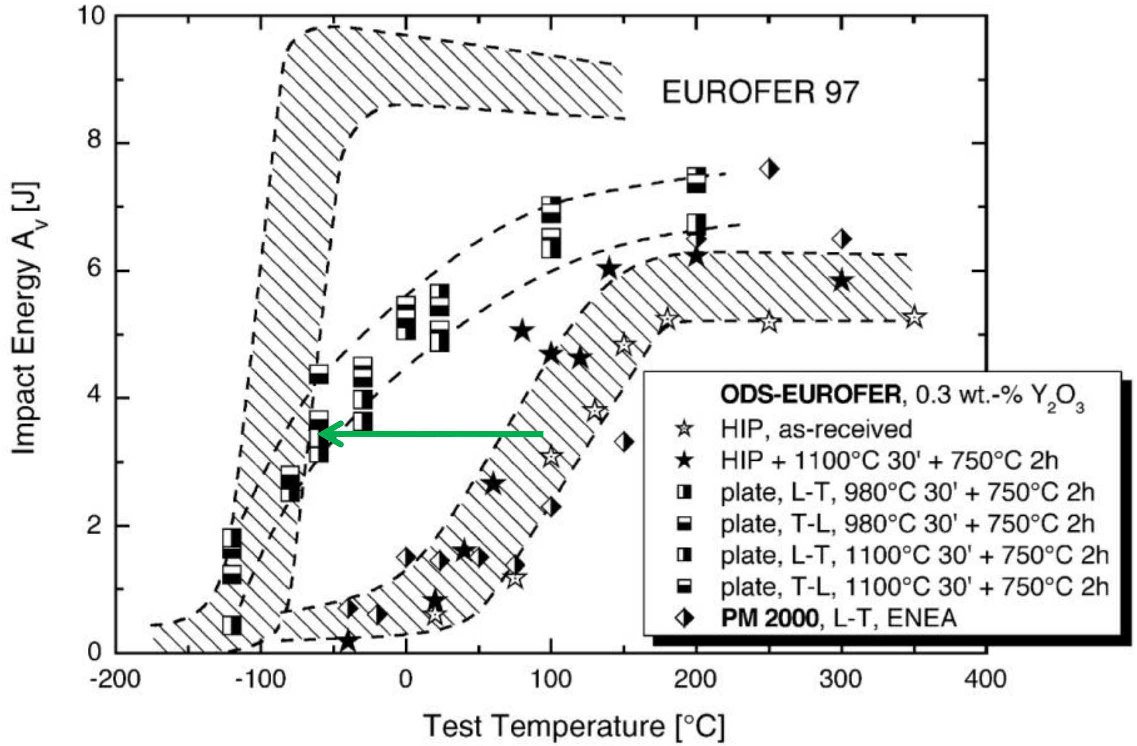
[1] ODS martensitic alloy for fuel pins for BOR-60 irradiation test (S.Ukai et al. Journal of NUCLEAR SCIENCE and TECHNOLOGY, Vol. 42, No. 1, 109–122, 2005)

[2] Corrosion resistance in supercritical pressurised water (N.Y.Iwata et al. *Materials Science Forum Vols. 654-656 (2010),166*

[3] Cladding for Gen IV SFR, CEA (Y. de Carlan et al. Journal of Nuclear Materials 386–388 (2009) 430–432)

ODS Ferritics: nuclear applications

Test temperature dependence of total absorbed energy of different ODS-EUROFER steels c.f. EUROFER 97

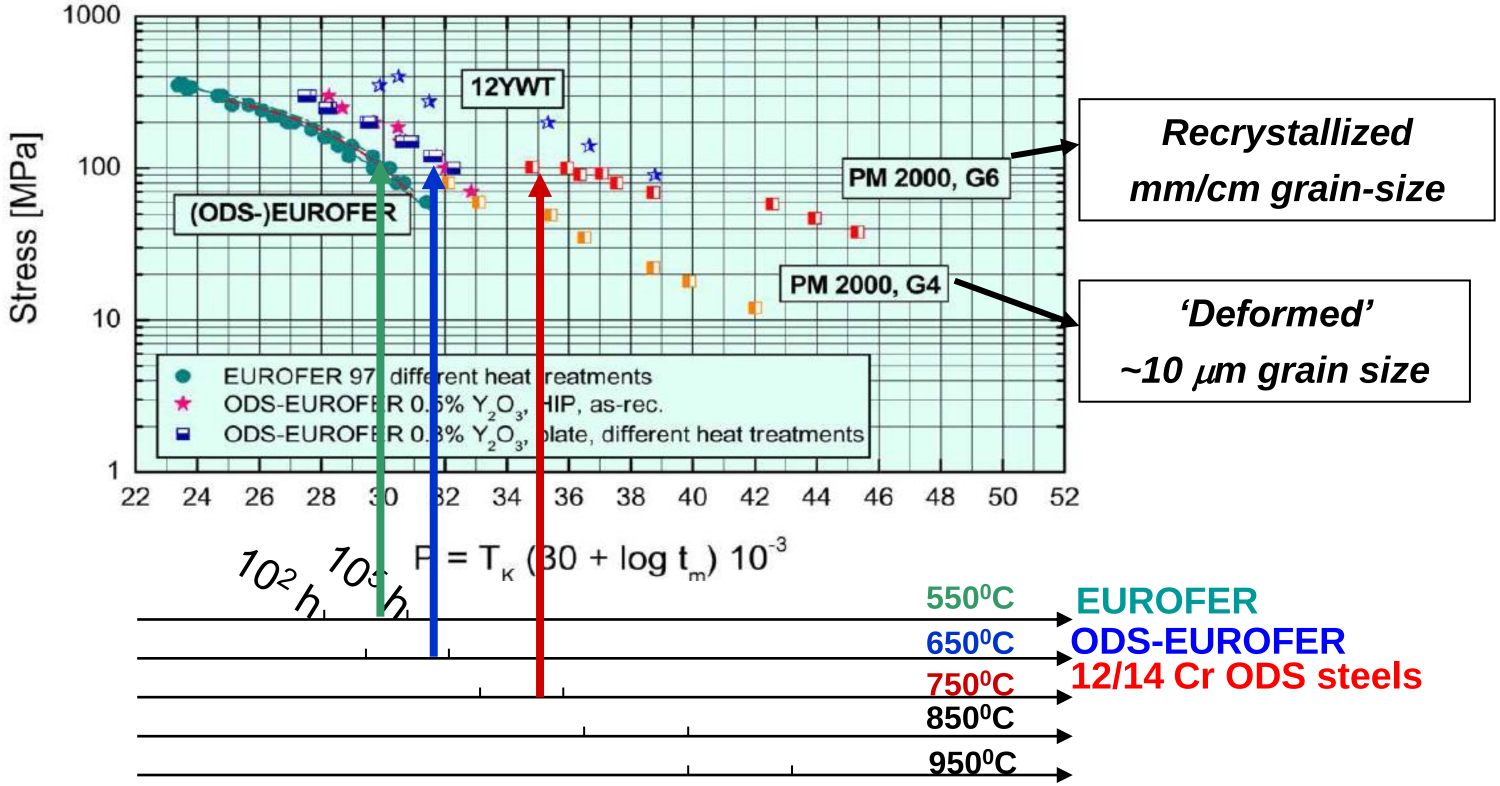


ODS EUROFER	DBTT
HIP/Heat treat	+60^{0}C/+100^{0}C
↓ HIP/Hot cross-roll/Heat treat	-80^{0}C/-40^{0}C

R. Lindau et al. , Fusion Engineering and Design 75–79 (2005) 989–996

ODS Ferritics: nuclear applications

Temperature Upper Limit Increase ODS F/M steels



Estimated Upper Limit on the Basis of a Creep strength of 100 MPa for ~50,000 Hours

Jean- Louis Boutard EFDA-CSU Garching, Nuclear Fission and Fusion Steels: Fundamentals and Applications.
UKAEA Culham Science Centre, Oxfordshire (UK), 8th & 9th of June 2009

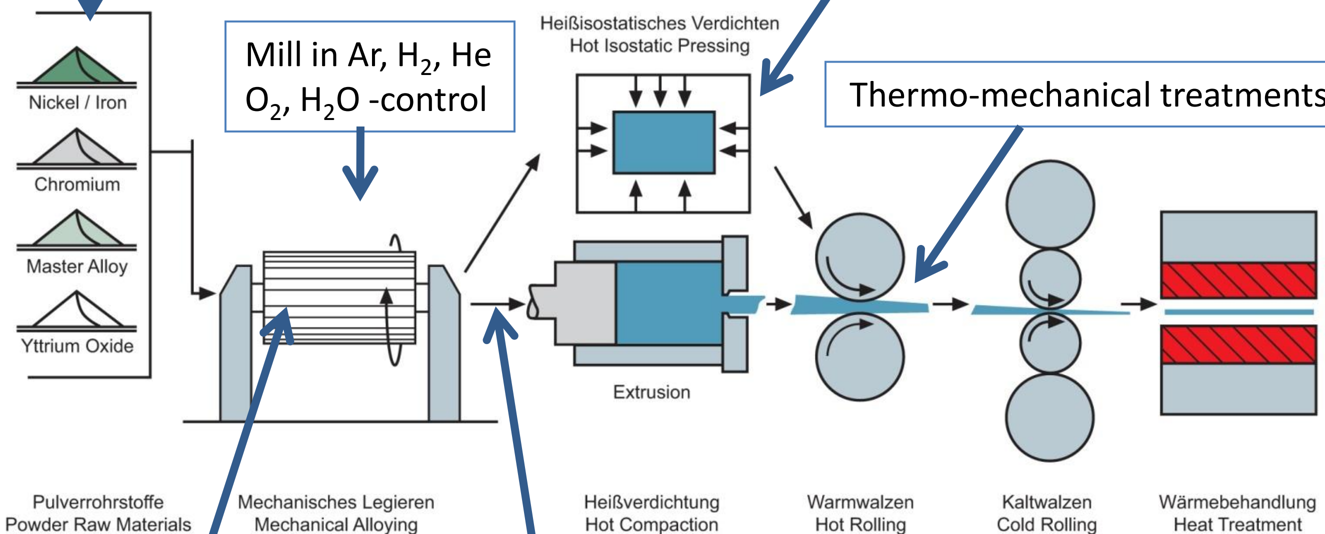
ODS Ferritics: nuclear applications

Elemental/Master alloy
Y_2O_3 , YFe_3
Blend powders pre-MA
Particle size range - constituent powder

HIP T^0C, time, p
Post-HIP heat treatment
HIP + roll etc
Vacuum hot pressing
High speed hydrostatic extrusion

Mill in Ar, H_2, He
O_2, H_2O -control

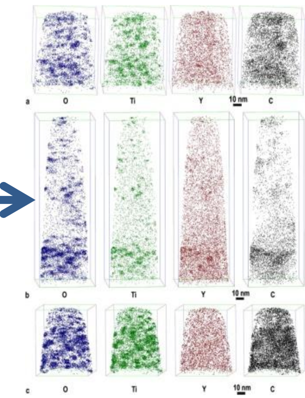
Thermo-mechanical treatments (various, multiple)



Mill type, speed, energy,
Contamination from mill materials (e.g. Al)
Ball sizes, size mixtures, composition
ball to powder ratios

Vacuum degass, T^0C, p, time
Gas shielded handling/storage

SANS
APT
HRTEM-FFT
EFTEM
HAADF
FIB



M.K. Miller et al., Journal of Nuclear Materials 351 (2006) 261–268

Historical Perspective: ODS alloy Development

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- ODS Ferritic alloys available for 40+ years

- Remained a niche product because of concerns over e.g.

  - Property anisotropy
  - Joining
  - cost

- A lot of work has now been done/significant results achieved suggesting property/processing/fabrication issues can be addressed

- Significant interest in these alloys remains (nuclear /fossil energy sector)

- New pathways to alloy production e.g. involving advanced gas atomisation techniques offer prospects of lower cost product forms