Evaluation of mechanical properties of ODS alloys in the GETMAT Project

Marta Serrano
Structural Material Division
CIEMAT

A. García-Junceda. M.Hernandez, F.J. Martin (CIEMAT), M. Rieth (KIT), R. Lindau (KIT), G. Müller (KIT), M. Yurechko (KIT), Y. de Carlan (CEA)
R. Chaouadi (SCK•CEN), M. Hoffman (MPA), C. Feuillette (MPA), P. Häner (JRC-IE), J. Chen (PSI), S. Concari (RSE), N. Luzginova (NRG), N. V. V. R. Kolluri (NRG)

2nd JOINT IAEA - EC TOPICAL MEETINGEC MEETING ON DEVELOPMENT OF NEW STRUCTURAL MATERIALS FOR ADVANCED FISSION AND FUSION REACTOR SYSTEMS

JRC Ispra (Italy) 16-20 April 2012

INTRODUCTION

- The GETMAT project (Generation IV and Transmutation Materials) (EU 7th FWP 2008-2013) coordinated by KIT
  - **Objective:** To integrate in a comprehensive and common effort within the European materials laboratories the R&D activities needed to select and characterize (in terms of mechanical behaviour, coolant compatibility and particle irradiation effects) structural materials with required properties for advanced nuclear reactor and transmutation systems.
  - **Participants:** KIT, ENEA, CNRS, Chalmers, MPA, VTT, Helsinski Univ, Univ Liverpool, HZDR, EDF, NRI, SCK-CEN, CEA, UPM, PSI, Univ. Alicante, CIEMAT, NRG, Univ. Libre Brux, Univ Edinburgh, CNR, KTH.
  - **Project cost:** 13.959.123 EURO, **Project Funding:** 7.500.000 EURO

- The objective of this presentation is to summarize the on going activities related to ODS characterization performed by the GETMAT partners

WP1 (CIEMAT): Metallurgical and mechanical behaviour

- **Materials Procurement, basic characterization and distribution (CIEMAT)**
- **Weld/Joining activities (KIT)**
- **HT Creep/Fatigue inert atmosphere (CIEMAT)**
- Data Base (JRC-IE)

WP2 (ENEA): Materials compatibility with coolant

- Corrosion tests in different media (Pb, He, SCW) (ENEA)
- Advanced corrosion barrier development (KIT)
- **Environmental effects on material mechanical properties (CIEMAT)**

WP3 (CEA): Irradiation behaviour

- BOITIX9, SUPERNOVA (PHENIX) (CEA)
- LEXUR (BOR 60) Pb, Gas (ENEA/SCK CEN)
- ASTIR (BR2) LBE, gas (SCK CEN)
- IBIS, SUMO (HFR) LBE, Na (NRG)
- STIP (SINQ) (PSI)
- MEGAPIE LBE (PSI)

WP4 (SCK CEN): Multiscale modelling and model experiments

- Modelling of fundamental properties of Fe and FeCr alloys (KTH)
- Modelling of radiation effects in Fe and FeCr alloys (CEA)
- Modelling-oriented experiments in Fe and FeCr alloys (CIEMAT)

MATERIALS

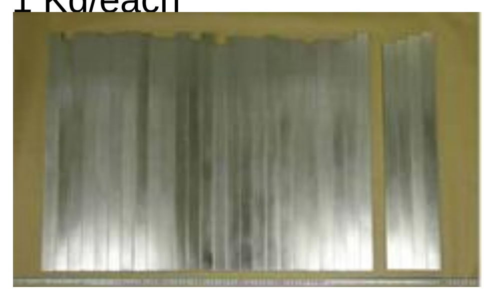
- 14Cr ODS - Fabricated and distributed by CEA
  - Fe 14Cr 1W 0.3Si 0.3Mn 0.15Ni-Ti + 0.3 Y_2O_3

Atomization → Attritor ball mill → Sealing → Hot extrusion 1100ºC → 1050ºC 1.5 h

3 bars (120mm x 45 mm diameter) of around 14 Kg/each

- 12Cr ODS - Fabricated by Kobelco and distributed by KIT
  - Fe 12Cr 2W 0.26 Ti + 0.23 Y_2O_3

29 plates (33x550x6.6 mm) of around 1 Kg/each

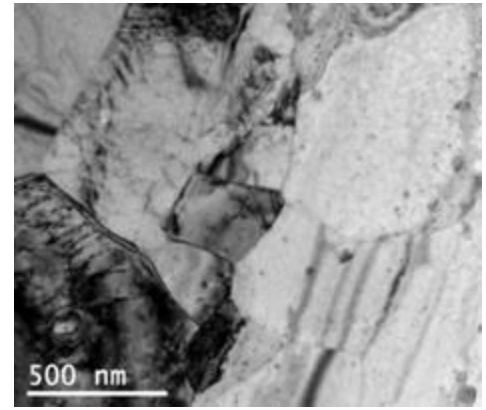


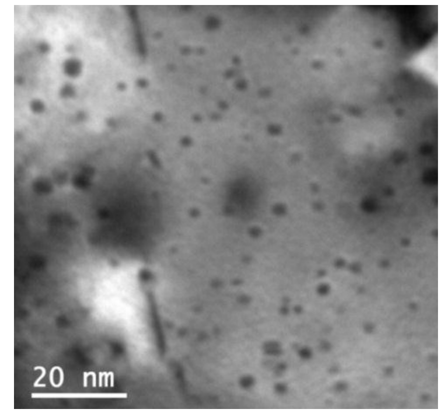
Atomization → Attritor ball mill → Sealing → Hot extrusion 1150ºC → Hot forging 1150ºC → 1150ºC 1 h → Machining → Cold rolling → 1200ºC 1 h

- 9Cr ODS – To be distributed in may 2012 by KIT
  - T91 + 0.3 Y_2O_3

Microstructure

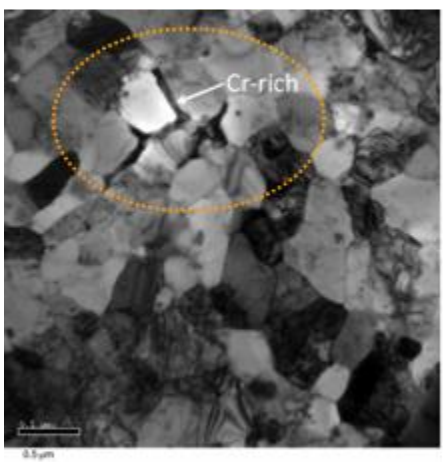
- 14Cr ODS Microstructure
  - The microstructure is constituted by small elongated grains (cigar shape) along the extrusion direction
  - Preferential crystallographic orientation of the grains along <110>// extrusion direction
  - Nano (Y-Ti-O) phases are uniformly distributed all over the grains.
  - Cr-rich phase decorating some grain boundaries

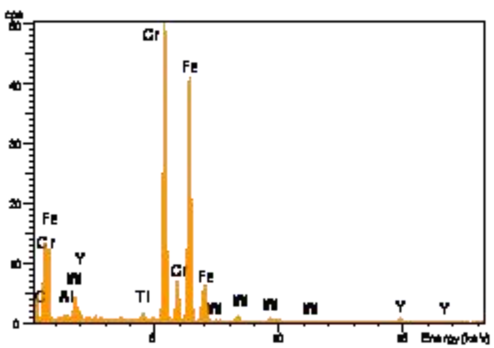


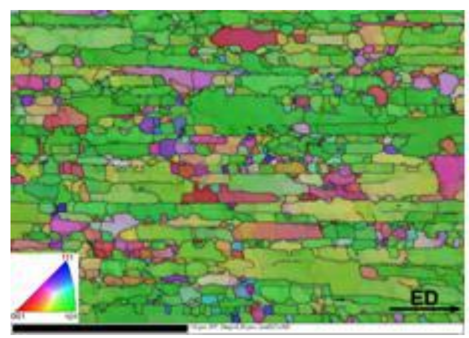


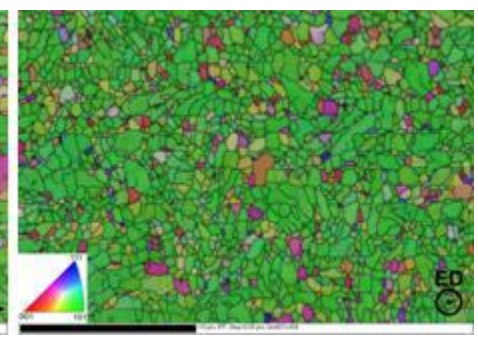
Ciemat
Centro de Investigaciones Energéticas, Medioambientales y Tecnológicas

Longitudinal			Transverse		
Length (nm)	Width (nm)	GAR	Length (nm)	Width (nm)	GAR
714 ± 18	421 ± 58	2.2 ± 0.2	438 ± 7	380 ± 18	1.7 ± 0.1





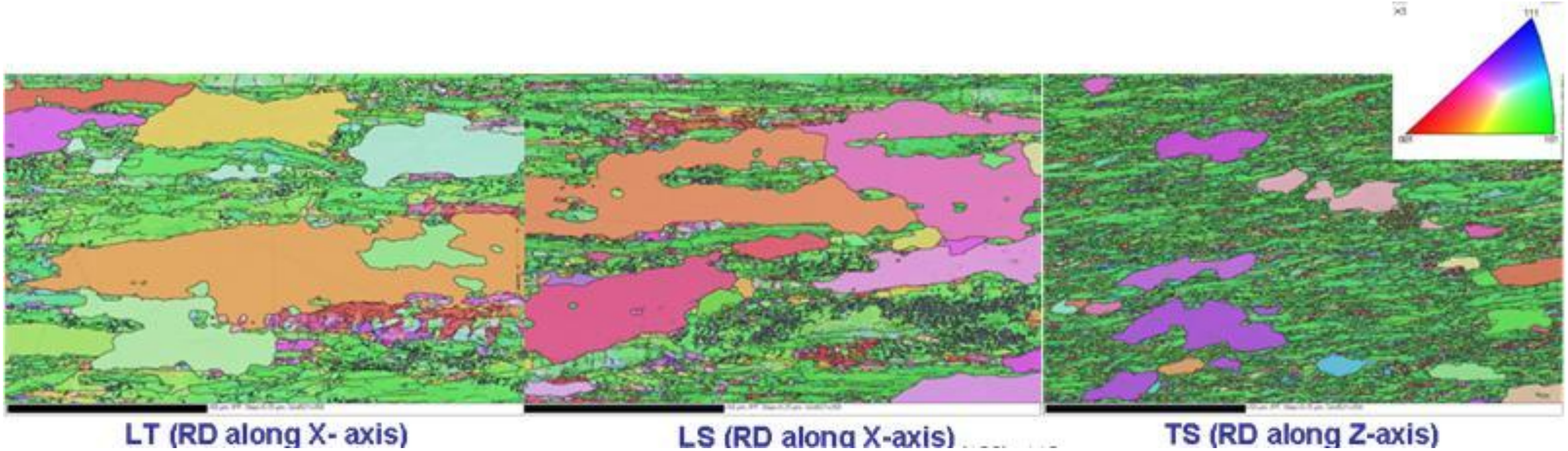




Microstructure

- 12Cr ODS Microstructure
  - More elongated microstructure in both longitudinal orientations, LT and LS, along the rolling direction.
  - Precipitations between 0.5 and 0.9 μm mainly consisting in Ti and O
  - Pores aligned in the rolling direction
  - Bimodal Grain Size: Small grains size < 1 μm heavily deformed and some elongated grains size up to 200 μm
  - The biggest grains have not got a preferential orientation.
  - The smallest grains seem to have a preferential orientation along <110> in the RD.

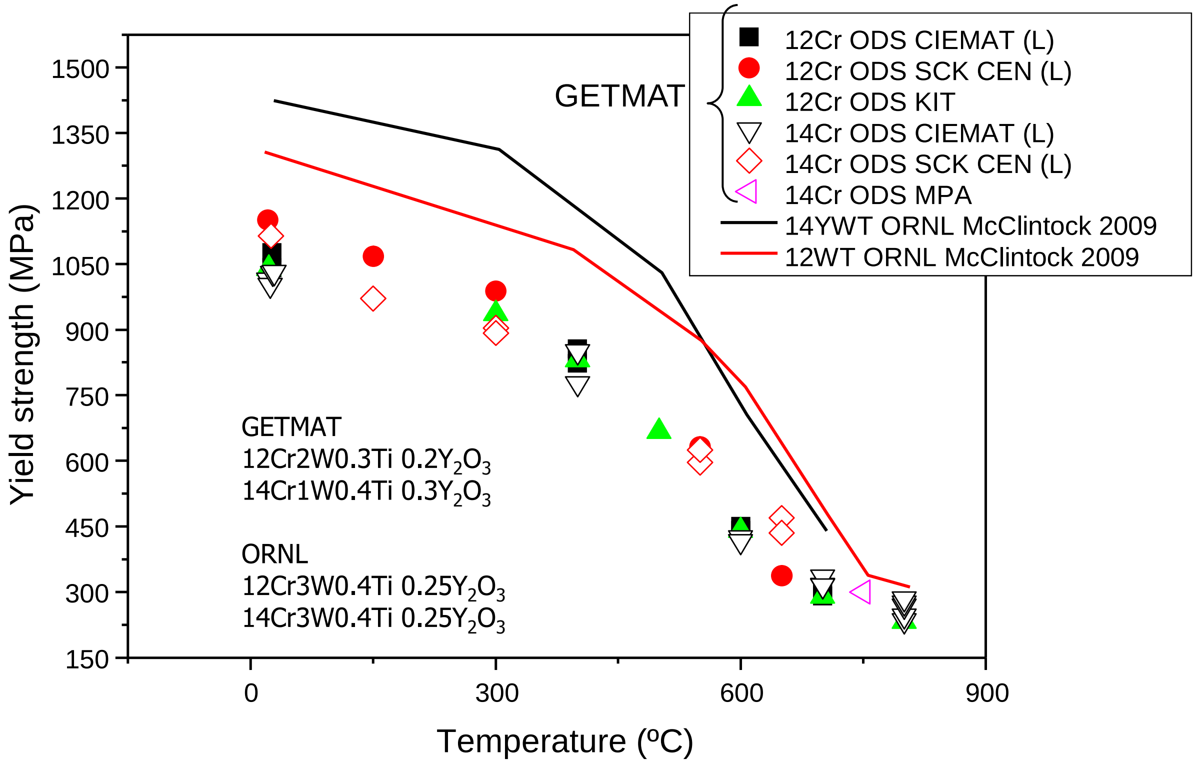
KIT Karlsruhe Institute of Technology



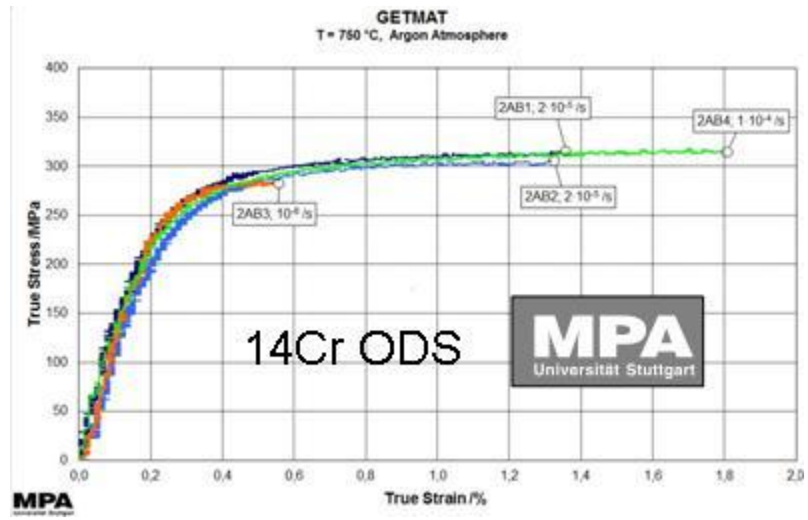
Ciemat
Centro de Investigaciones Energéticas, Medioambientales y Tecnológicas

Tensile properties

- Yield strength is slightly lower for GETMAT ODS alloys than for 14YWT and 12YWT
- No effect of Cr content on YS - It was expected a slight lower YS for 12Cr ODS alloys
- 14Cr ODS - Very low ductility (0.54 to 1.9%) at 750ºC for strain rates varying between 1.10^{-4} and 10^{-6} s-1
- 12Cr ODS - A maximum in elongation at 600ºC

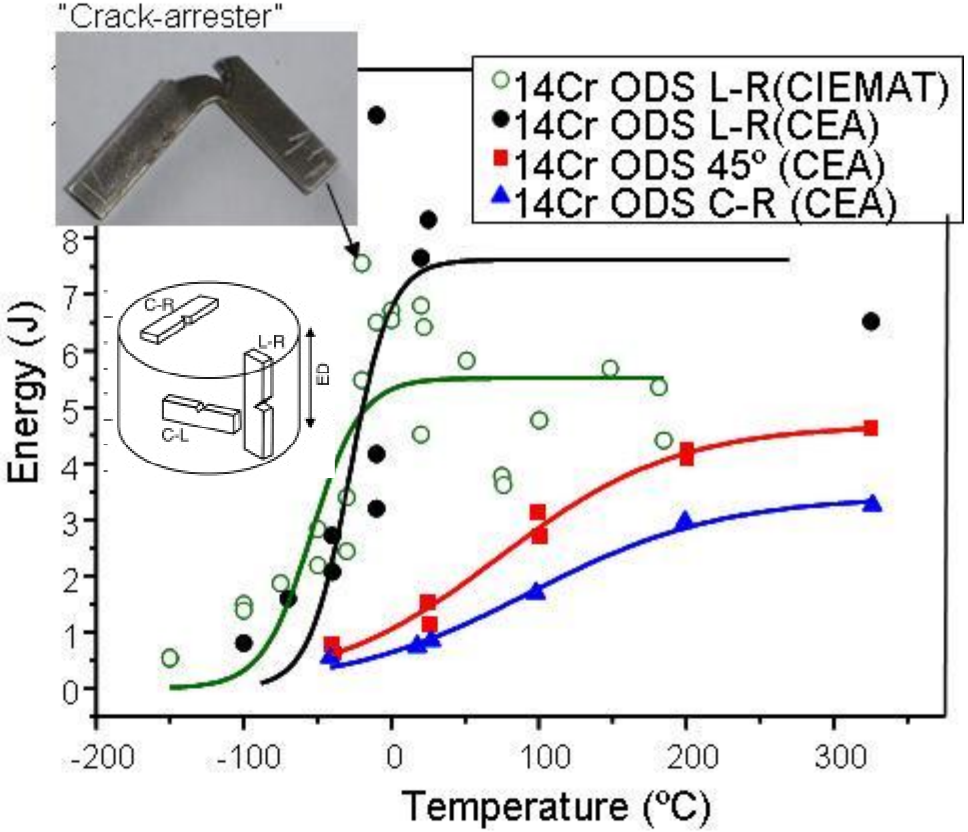


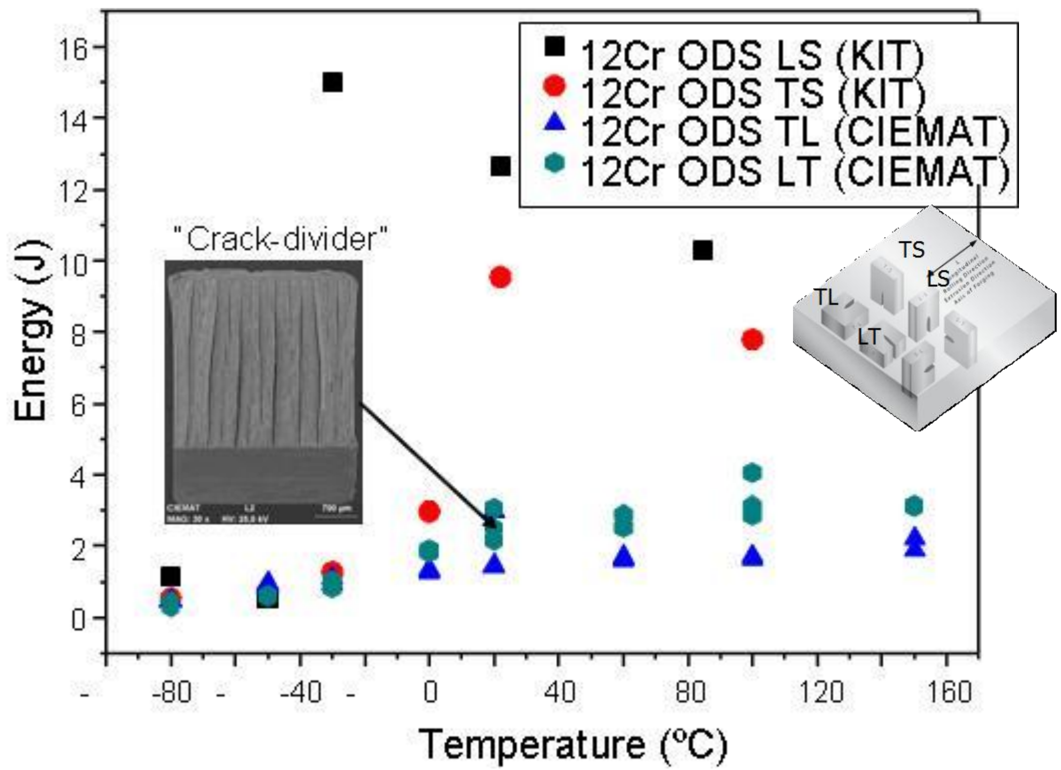
Both alloy show anisotropy:
- Transverse oriented specimens shows lower ductility than longitudinal oriented ones.



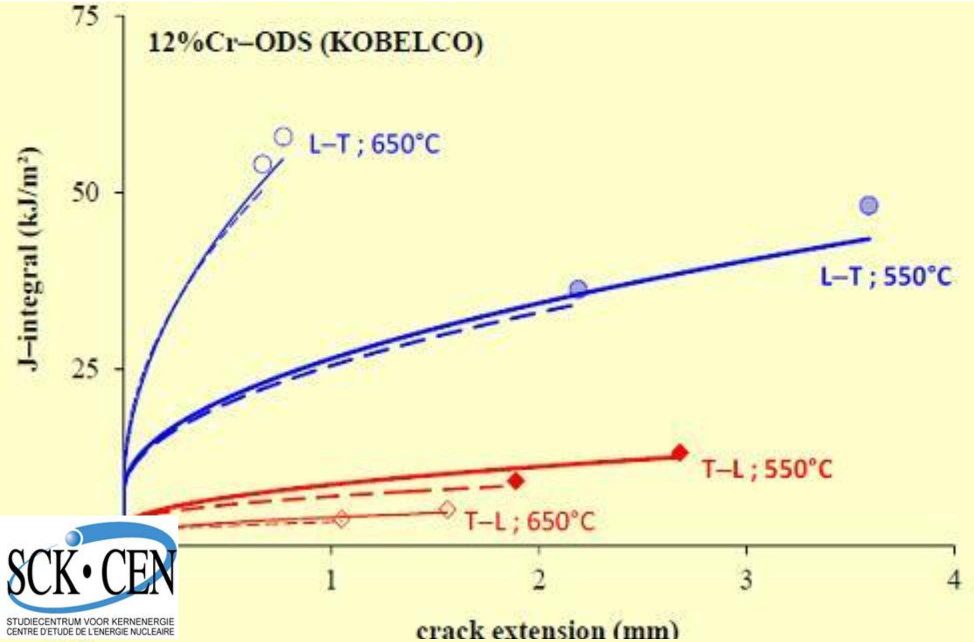
Impact properties

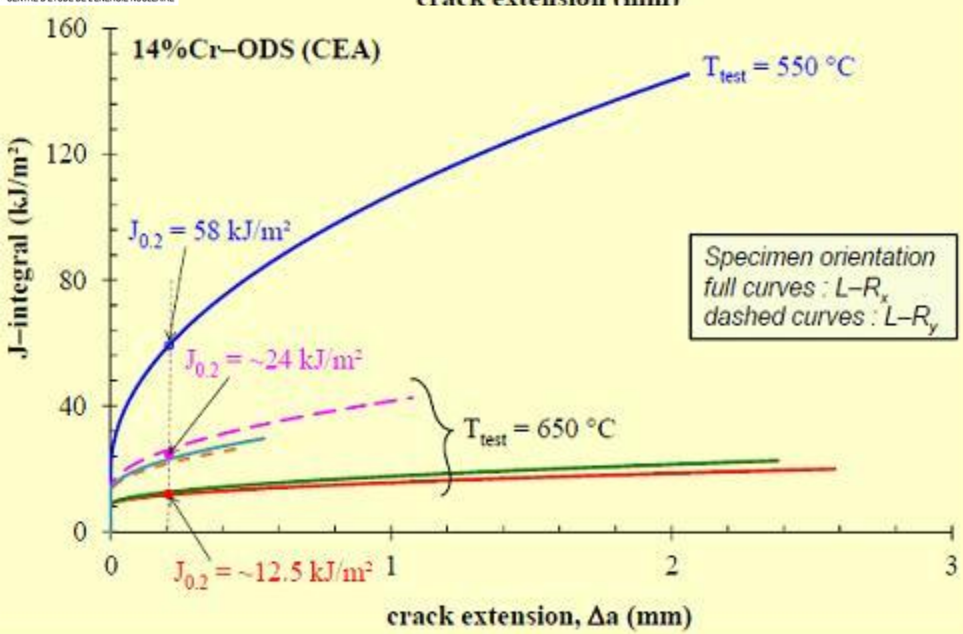
- Higher USE due to toughening effect of delaminations in both CEA and CIEMAT LT specimens
- Low USE and high DBTT for the specimens in the CL and 45º orientation
- Similar to other ODS alloys



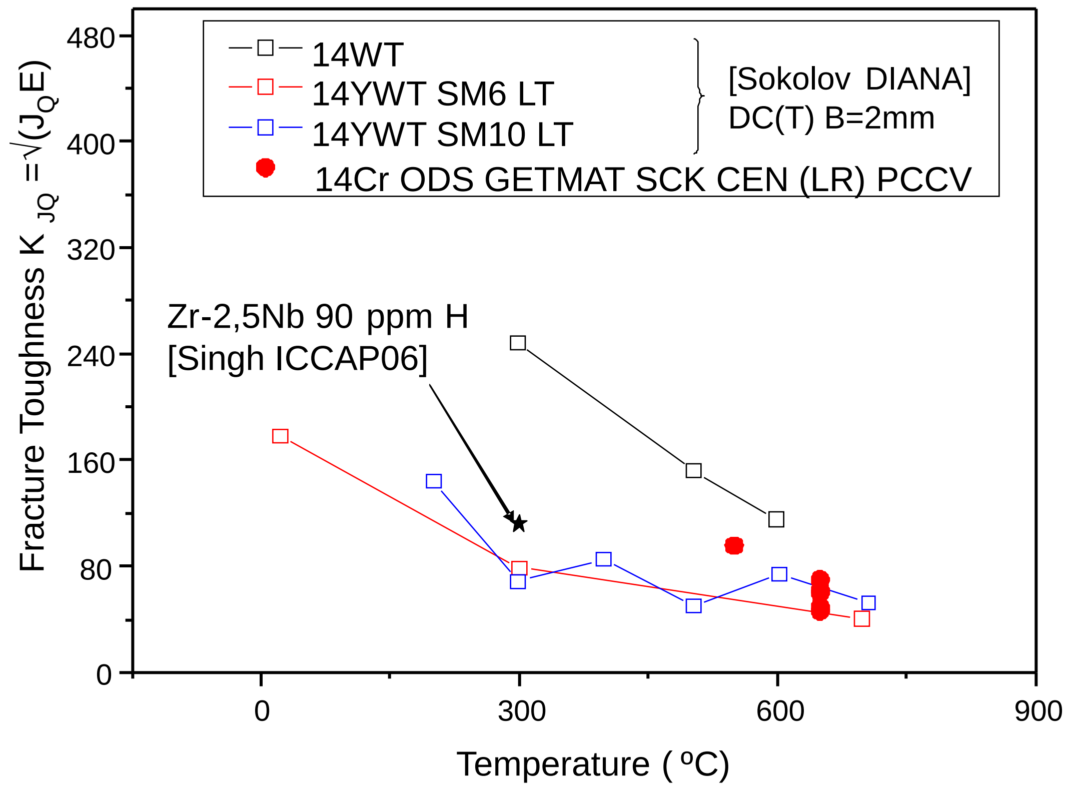


Fracture toughness properties



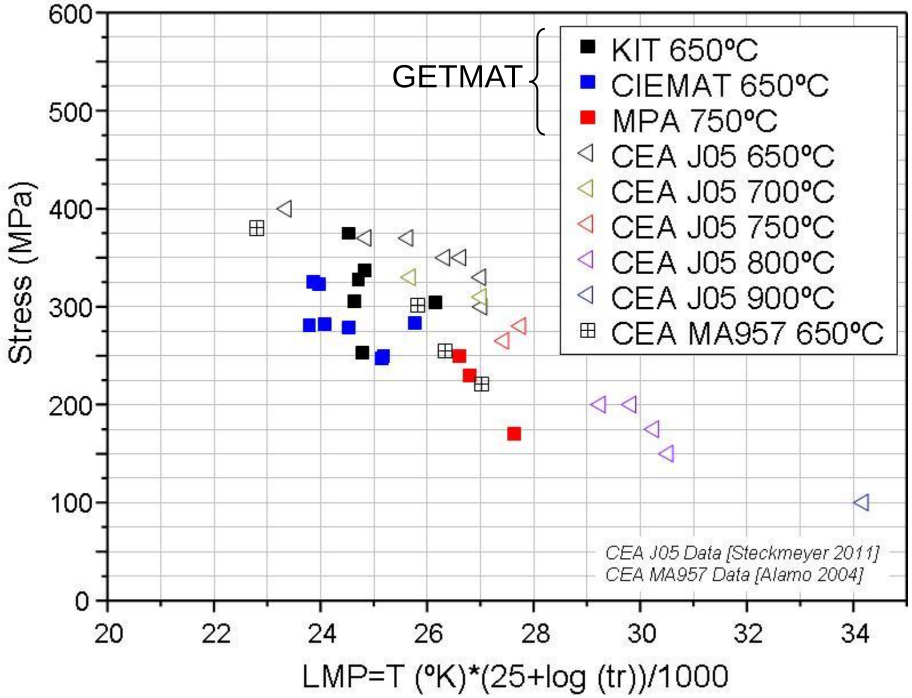


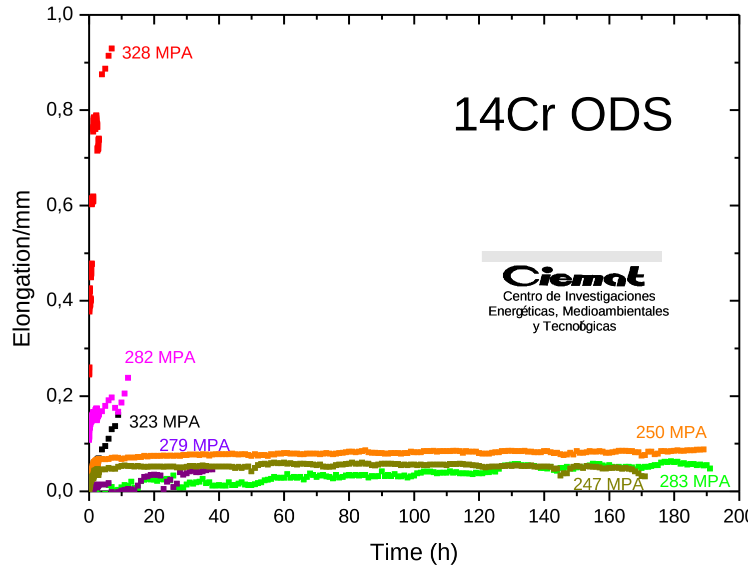
- High temperature (>550 °C) crack resistance of high Cr–ODS steels is weak.
  - 14Cr ODS Similar to 14YWT and Hydrided Zr-2.5Nb
- Comparison to standard (non–ODS) high Cr–steel clearly shows drastic loss of crack resistance (10 to almost 100 times lower).



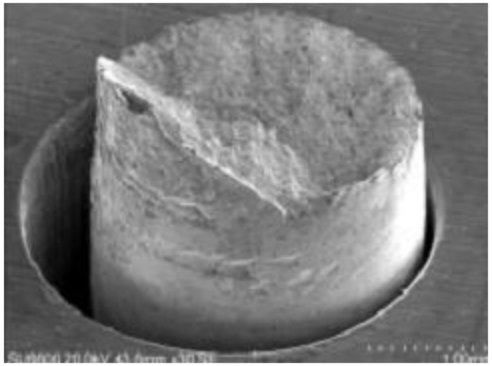
Creep properties

- No tertiary creep
- Rupture time lower than expected. The scatter is quite high
- Brittle fracture

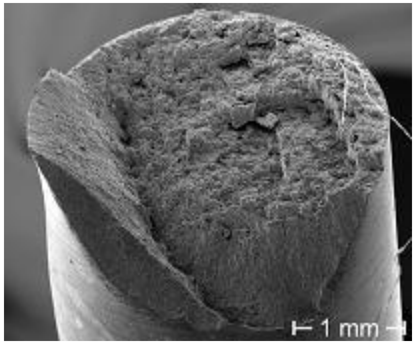




CIEMAT 650ºC 328 MPa



KIT 650ºC 337 MPa

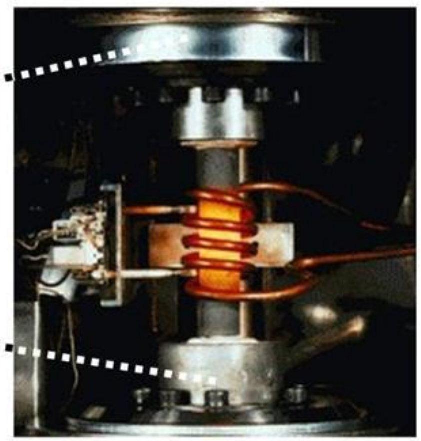


Low Cycle Fatigue

- Test to be performed by JRC-IET and MPA

Testing machine

MTS-TT (tension-torsion) facility with vacuum chamber and induction heating system



MPA Test matrix

Material	Test type	T (°C)	Strain rate (%/s)	Total train range (%)	HT Tens. (min)	Target duration (cycles)	Target duration (h)	Status
14%Cr ODS	Pure Fatigue	750	1,00E-03	to be defined	0	500		-
14% Cr ODS	Pure Fatigue	750	1,00E-03	tbd	0	2000		-
14% Cr ODS	Pure Fatigue	750	1,00E-03	tbd	0	10000		-
14% Cr ODS	Pure Fatigue	750	1,00E-03	tbd	0	30000		-
14% Cr ODS	Creep-Fatigue	750	1,00E-03	tbd	10		100	-
14% Cr ODS	Creep-Fatigue	750	1,00E-03	tbd	10		1000	-

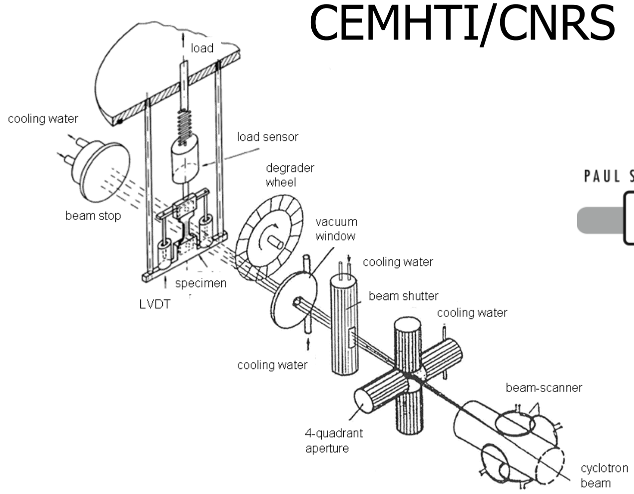
Material	Test type	T (°C)	Strain rate (%/s)	Total train range (%)	HT Tens. (min)	Target duration (cycles)	Estimated duration (h)	Status
9%Cr ODS	Pure Fatigue	600	1,00E-03	to be defined	0	500		-
9%Cr ODS	Pure Fatigue	600	1,00E-03	tbd	0	2000		-
9%Cr ODS	Pure Fatigue	600	1,00E-03	tbd	0	10000		-
9%Cr ODS	Pure Fatigue	600	1,00E-03	tbd	0	30000		-
9%Cr ODS	Creep-Fatigue	600	1,00E-03	tbd	10		100	-
9%Cr ODS	Creep-Fatigue	600	1,00E-03	tbd	10		1000	-

Material	Test type	T (°C)	Strain rate (%/s)	Total train range (%)	HT Tens. (min)	Target duration (cycles)	Test duration (h)	Status
12%Cr ODS	Pure Fatigue	650	1,00E-03	tbd	0	2000		-
12%Cr ODS	Creep-Fatigue	650	1,00E-03	tbd	10		100	-

In beam creep

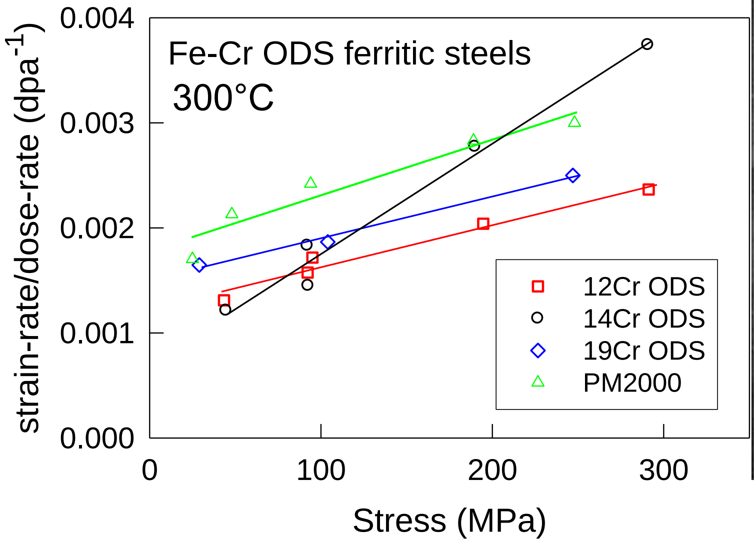
- Irradiation creep rates of both 12Cr and 14Cr -ODS ferritic steels show linear stress dependence up to 300 MPa at a temperature of 300°C. Irradiation compliance amounts to 4.0x10-6 dpa-1·MPa-1 and 10x10-6 dpa-1·MPa-1 at a temperature of 300°C for 12Cr and 14Cr -ODS, respectively.
- No remarkable effects of neither Cr content, nor grain size or dispersoid size on irradiation creep properties are observed.
- Dislocation loops and helium bubbles are distributed homogenously in the matrix. In the case of high density fine dispersoids, most bubbles are attached to ODS particles..

under cooperation between PSI and CEMHTI/CNRS

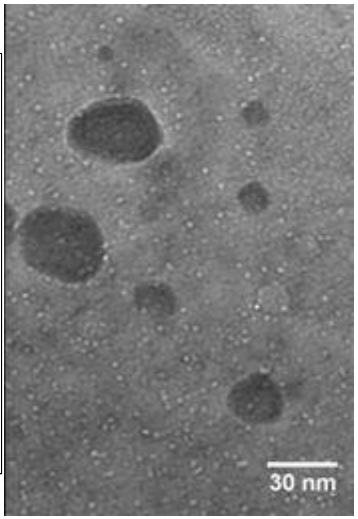


PAUL SCHERRER INSTITUT

26MeV alfa-particles

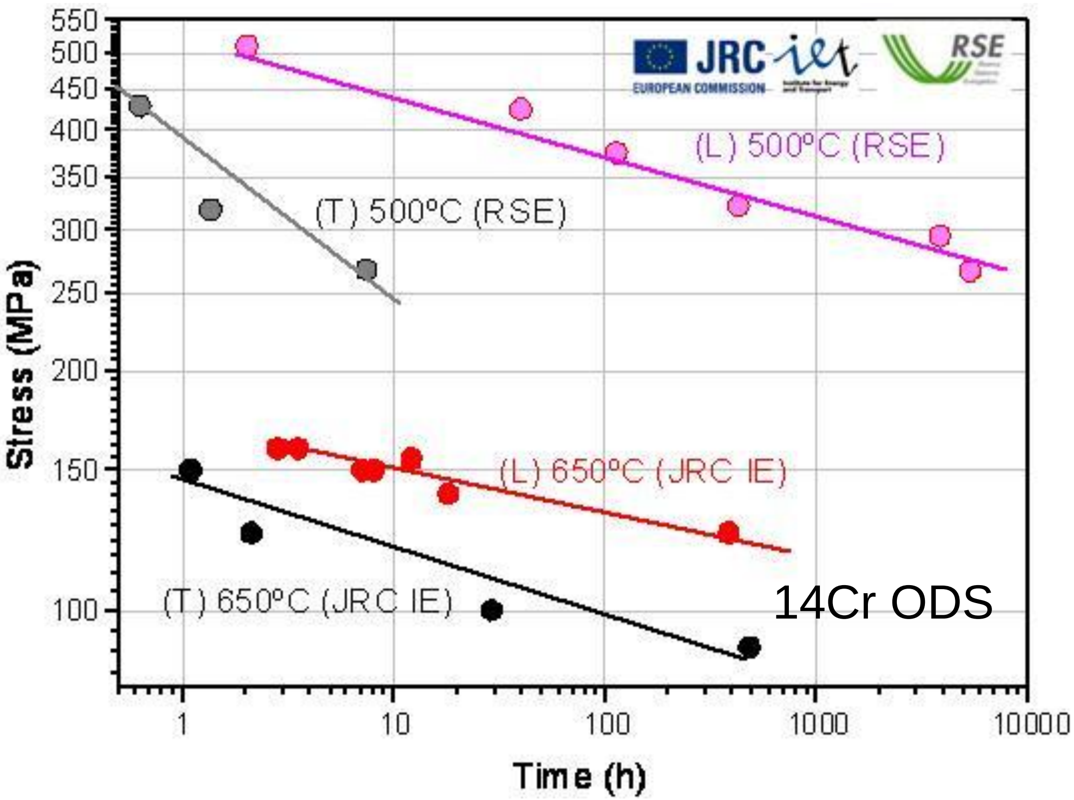


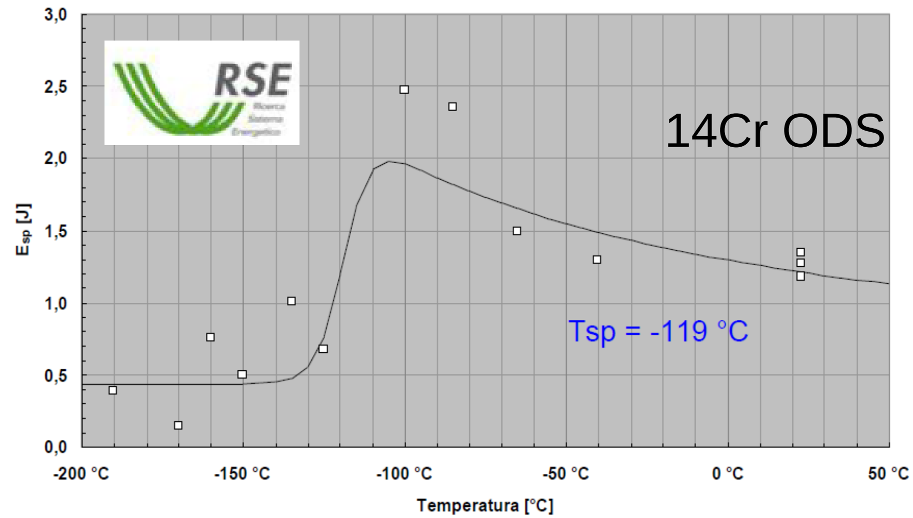
14Cr ODS
He-implantation 300°C

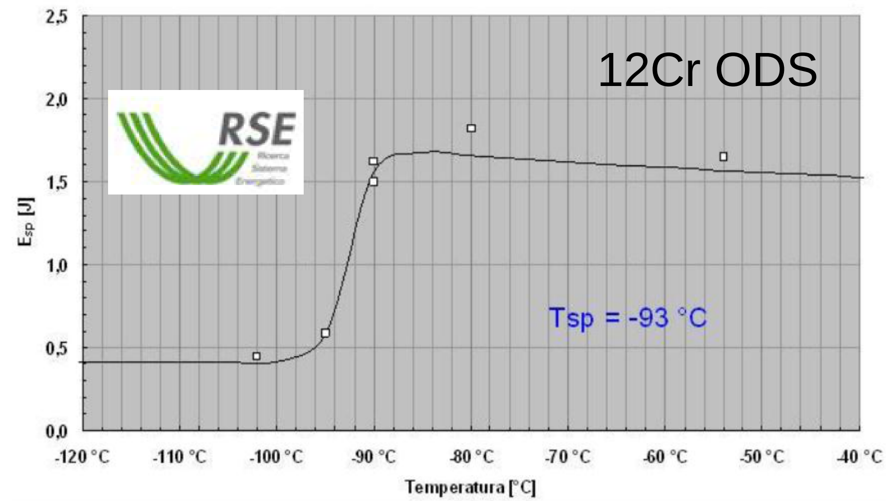


Small punch properties

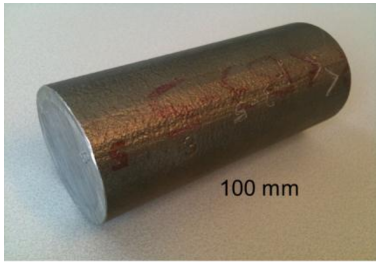
- Creep-rupture time is lower for the specimens in the transverse orientation
- Transition temperature is lower for 14Cr ODS – same tendency as KLST tests

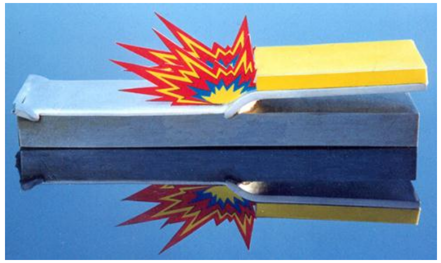




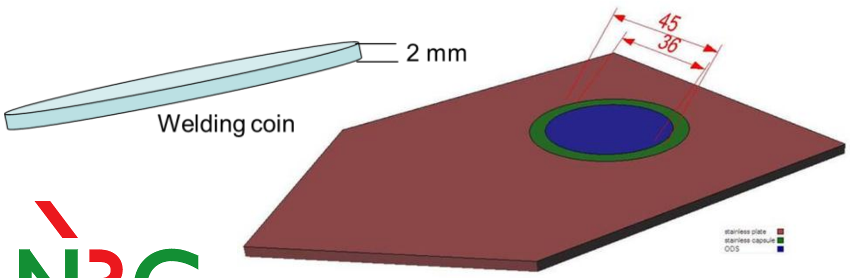


Welding/Joining Methods



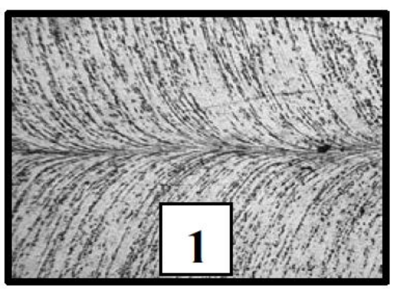


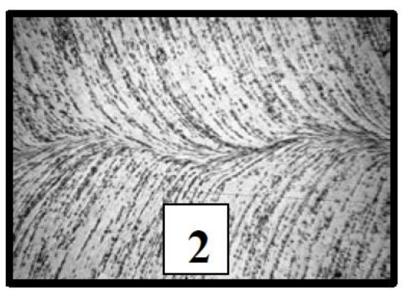
As received material



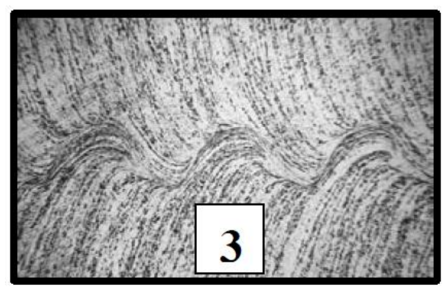
Welding set-up

- 14Cr ODS steel (supplied by CEA) has been successfully joined by explosive welding techniques.
- It have been determined that 14Cr ODS steel has a wide weldability window.
- Due to extremely high strength and low ductility, the explosive welding of 14Cr ODS material should be done with pre-heating to 200oC and by applying higher impact velocity (explosive layer height) compared to the conventional steel.

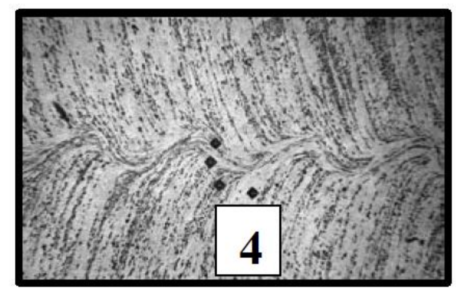




Lower limit: *no waves/high shear strain rate*



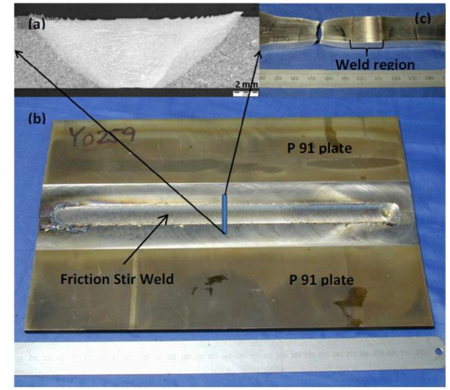
Best conditions: *regular waves*



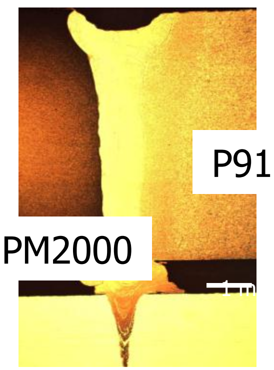
Upper limit: *extra deformation/ smaller waves/ "pocket" formation*

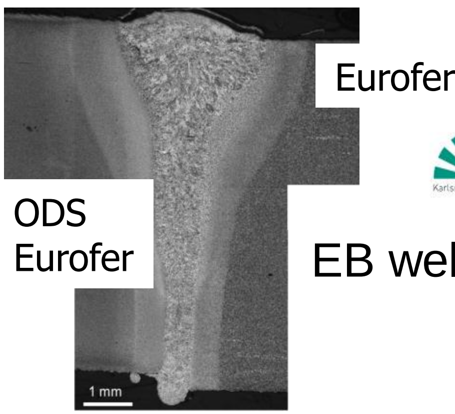
Welding/Joining Methods

- Other methods:
  - Friction stir welding /NRG) – Linear friction stir welding on 12Cr ODS
  - Electro-Magnetic Pulse technique (JRC-ITU) – P91 and P92
  - EB ang TIG (KIT) – Eurofer, Eurofer ODS, P91, P92, PM2000
  - Diffusion bonding (KIT) – PM2000

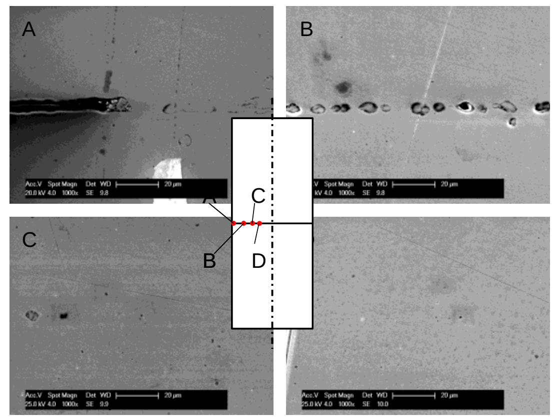


Friction stir welding

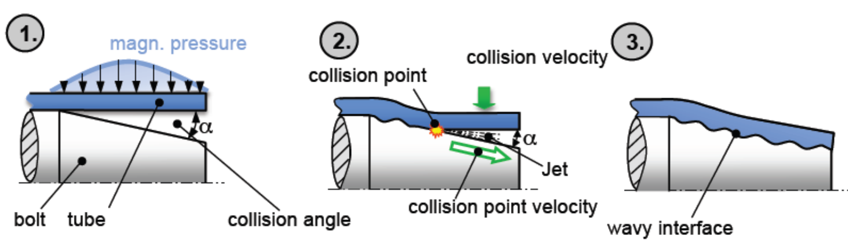




EB welds



Diffusion bonding PM2000



Electro-Magnetic Pulse technique

Summary

- One of the GETMAT objectives is to fabricate and characterize ODS alloys to be use as cladding of Gen IV reactors
- Short term and long term mechanical properties are on going for a 14Cr ODS (CEA) and 12Cr ODS (KIT)
- The behaviour of both alloys is as expected:
  - Homogeneous distribution of nano-oxydes inside the grains
  - Mechanical properties depends on the orientation of the samples - due to the grain morphology and grain boundary precipitates.
  - High resistance at high temperature
  - Low fracture toughness at high temperature
- Some preliminary results on joining method are available
- Compatibility of 14Cr ODS and 12Cr ODS with liquid Pb, Supercritical water and impure Helium is included in the project

Next steps

- On-going activities
  - Long term tests and thermal ageing experiments
  - Irradiations are on going and some results will be available at the end of this year
  - Characterization of welds

The Joint GETMAT-MATTER International School on Materials UNder Extreme COnditions (MUNECO)

June 11-15, 2012

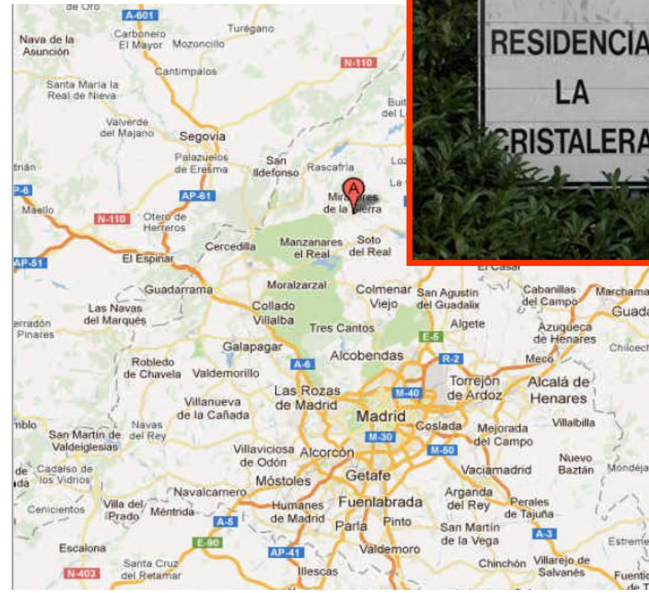
“La Cristalera” en Miraflores de la Sierra
Madrid, Spain

Organisers:
P. Agostini, ENEA (coordinator MATTER)
C. Fazio, KIT (coordinator GetMat)
M. Serrano (CIEMAT)
M. Utili (ENEA)
J. Van den Bosch (SCK·CEN)

Web page: http://www.matterfp7.it/ → training

MUNECO Venue

- The conference will take place at La Cristalera, the meeting center of Universidad Autonoma de Madrid, located in Miraflores de la Sierra, about one hour by car from Madrid.
- **40 rooms**
- **Meeting rooms**
  - 1 conference hall 90 people
  - 1 meeting room 70 people
  - 2 small meeting rooms for 25 people.
- Accommodation of speakers: Hotel at Miraflores. Shuttle bus
- La Cristalera has several **recreational facilities**, for basketball, football and tennis. There's also a swimming pool and several interesting hiking paths.



MUNECO registration info

- The MUNECO International school only admit a **limited number of participants (max 40.)**
- A fee of **200 euros** will include book of abstracts, CD, accommodation, lunches and dinners, social activities and banquet
- Participants are encouraged to submit an abstract (max DIN A4 page) describing their current research results.
- The abstract will be used as a text basis for the review process:
  - All applications will be reviewed to find a fair distribution of the available places among all qualified applicants from the various countries and institutions. In case of conflict, preference will be given to the participants who filed their application earlier
- If your submission is accepted, you will also have to prepare and present a poster during the summer school.
- Important deadlines
  - **27th April: Abstract Submission**
    - ✓ **Marta.serrano@ciemat.es**
  - **4th May: Notification of acceptance**
  - **25th May: Fee payment**

M	T	W	T	F	S	S	
			1	2	3	4	
5	6	7	8	9	10	11	March
12	13	14	15	16	17	18	
19	20	21	22	23	24	25	
26	27	28	29	30	31	1	
2	3	4	5	6	7	8	
9	10	11	12	13	14	15	April
16	17	18	19	20	21	22	
23	24	25	26	**27**	28	29	
30	1	2	3	**4**	5	6	
7	8	9	10	11	12	13	
14	15	16	17	18	19	20	May
21	22	23	24	**25**	26	27	
28	29	30	31	1	2	3	
4	5	6	7	8	9	**10**	Jun
11	**12**	**13**	**14**	**15**	16	17	