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Detailed work plan and testing matrices

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Summary

This report includes the details of the materials distributed within WP4 that consist of two ODS bars (14Cr and 9Cr) and three ODS cladding tubes (9Cr, 14Cr and 18Cr ODS). All the materials were fabricated and distributed by CEA. The proposed test matrix is also included.

Approval

Rev.	Date	First author	Project Coordinator
0	July/2014	Marta Serrano (CIEMAT) 21-July-2014	Aurore Michaux (CEA)

Distribution list

Name	Organisation	Comments
All beneficiaries	MatISSE	

Table of contents

1 Objectives

The objective of this deliverable is to compile the main characteristics of the ODS materials distributed within MatISSE WP4 and to summarize the proposed test matrix.

2 Introduction

The objective of this MatISSE WP4: “Characterization of ODS alloys for LFR and SFR cladding” is to contribute to the pre-designing of ODS alloys to be used as cladding for fast nuclear reactors, taking into account most of the research and developments that are being carried out in the European projects as well in the national programs of the different participants

Despite the fact that within GETMAT and MATTER FP7 projects no ODS tubes were within their scope, the experience gained on the characterization of plate and/or bars within these projects and several national programmes is considered very valuable to reach the objective of this Work Package. Along with new activities, the materials and experience from GETMAT and MATTER will be an integrated part of the MatISSE work package on ODS.

The WP is divided into three tasks:

- Task 4.1 - Role of the microstructure on the mechanical behaviour (HZDR)
- Task 4.2 - Characterization of ODS cladding tubes (VTT)
- Task 4.3 - Characterization of ODS under safety-related operating conditions (CEA/KIT-IHM)

During a meeting held at CIEMAT on 6th March 2014 it was decided to focus task 4.1 activities on ODS bars with the same chemical composition and fabrication procedure as those bars used to fabricate the ODS cladding tubes to be characterized within task 4.2. Two cladding tubes, 9Cr-ODS and 14Cr-ODS will be distributed for task 4.2 activities (microstructure and mechanical properties) and task 4.3 activities (characterization under temperature and pressure transients, PSI). The distribution will be done in two steps. The first delivery will prioritize those partners that are going to carry out creep experiments and thermal ageing treatments. A second delivery will be done next year for the remaining partners. Around 1.8 meters of each tube (9Cr-ODS and 14Cr-ODS) are available for the first delivery, and it is expected to have a similar quantity for the second step. In addition a 18Cr-ODS cladding tube is going to be distributed for plugging trials of pressurized tubes and possibly for burst tests. Regarding task 4.3 “corrosion/oxidation activities” it was decided to use the 9Cr-ODS plates and 12Cr-ODS plates from GETMAT project that are located at KIT.

3 ODS bars

3.1 GETMAT 14Cr-ODS bar

The 14Cr ODS bar was supplied by CEA within the GETMAT project (the code for this material was J27-M2). The pre-alloyed powder was produced by Aubert & Duval France by gas atomization. Mechanical alloying of the pre-alloyed powder of composition (in wt%) 13.98Cr, 1.03W, 0.39Ti, 0.29Mn, 0.32Si and 0.17Ni with 0.3Y2O3 particles was performed at Plansee Austria under hydrogen atmosphere within a vertical attritor ball mill. The consolidation was carried out by hot extrusion at 1100ºC at CEFIVAL France. Finally, the manufactured bars were annealed at 1050ºC for 1.5h. The average chemical composition obtained on the final material was (in wt%) 13.5Cr, 0.22Y, 0.9W, 0.4Ti, 0.27Mn and 0.32Si.

Nano (Y-Ti-O) phases are uniformly distributed all over the grains. The microstructure of the 14Cr ODS bar shows an elongated-grain structure parallel to the extrusion direction, as seen in Figure 1. In the longitudinal plane, the material exhibits a bimodal grain size distribution, in which the smallest grains have a distribution ranging from 0.1 to 3.0 microns, representing a volume fraction of 80%, whereas the largest and more elongated grains have sizes between 3.0 and 9.0 microns with a volume fraction of 20%. By contrast, the in the transverse plane a more uniform distribution of grain size is observed, with a mean length of 432 ± 55 nanometres A preferential crystallographic orientation of the grains along <110> parallel to the extrusion direction is observed.

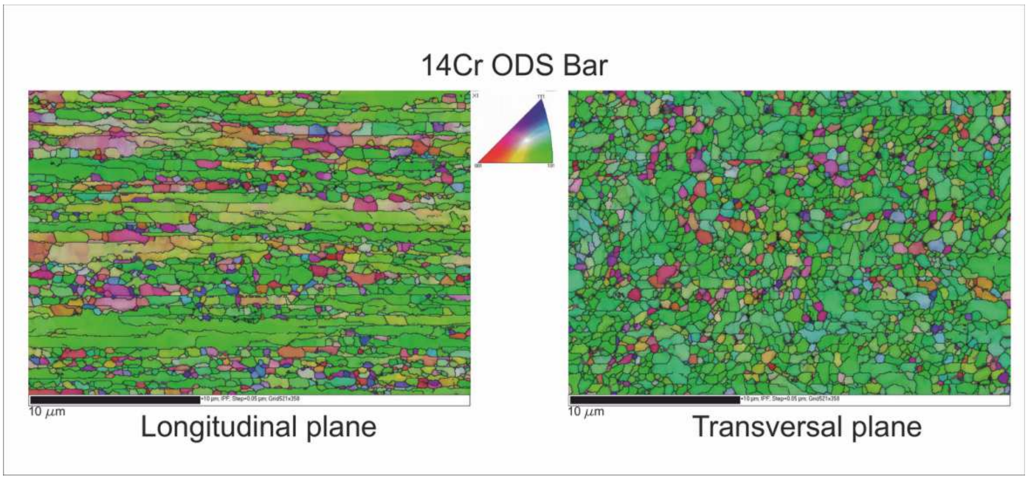

Figure 1.- Microstructure of the GETMAT 14Cr ODS bar

The tensile properties generated within GETMAT project can be seen in Fig. 2.

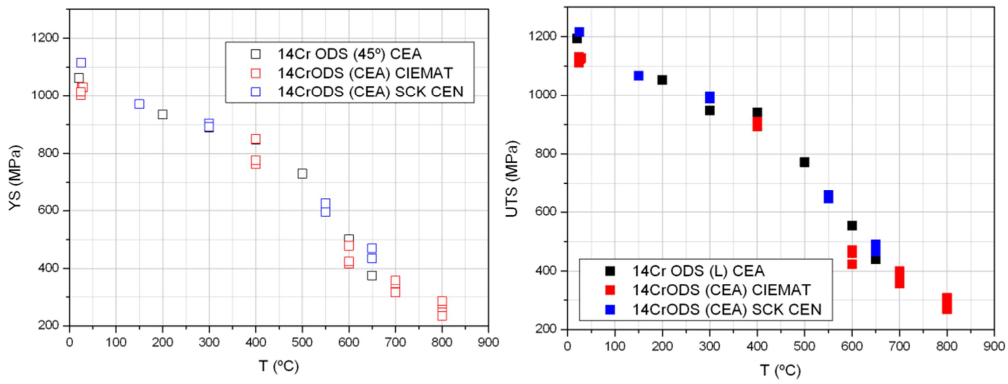

Figure 2.- Tensile properties of the GETMAT-14Cr-ODS bar

The available amount of 14Cr-ODS (GETMAT) bar (diameter 36 mm) is very limited. Pieces of bars or remnants will be cut by CIEMAT and sent to CNRS, EDF and KIT (IAM-AWP). Remnants of the same bar with missing inner part will be cut by HZDR .

3.2 MATISSE 9Cr-ODS bar

The Fe9Cr ODS Bar (SRMA code : L22-M1) is an extruded bar in the ferritic state. Pay attention, in order to correctly simulate the behavior of the Fe-9Cr ODS tube it is important to homogenize the machined samples at 1050°C with a very efficient quench (10°C/s) and an annealing at 750°C. Indeed the critical speed to get a martensitic structure (like for the tubes) is very high (see for example the CCT diagram on a similar alloy).

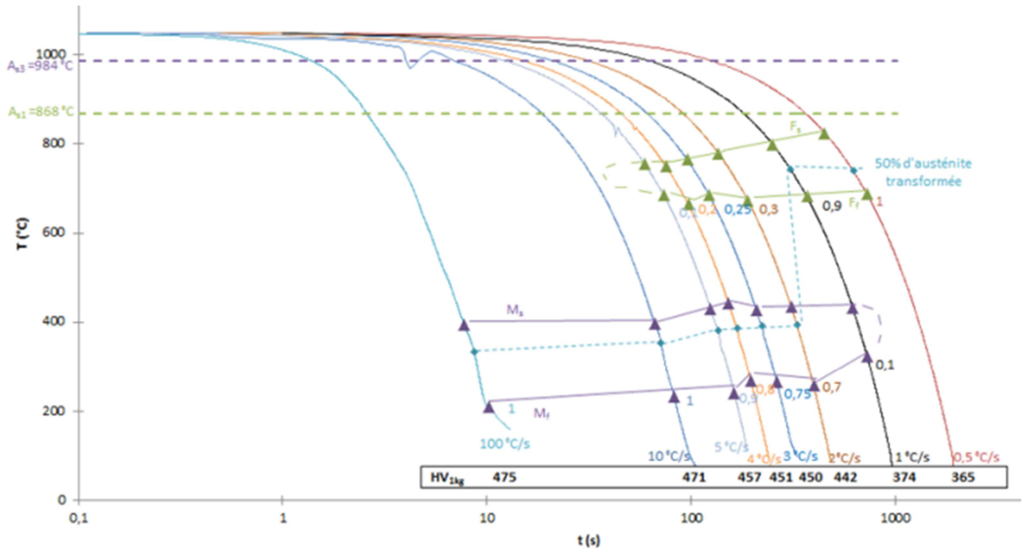

Figure 3.- CCT diagram of a 9Cr ODS alloys

The share of 9Cr-ODS bar among the partners can be seen in Figure 4.

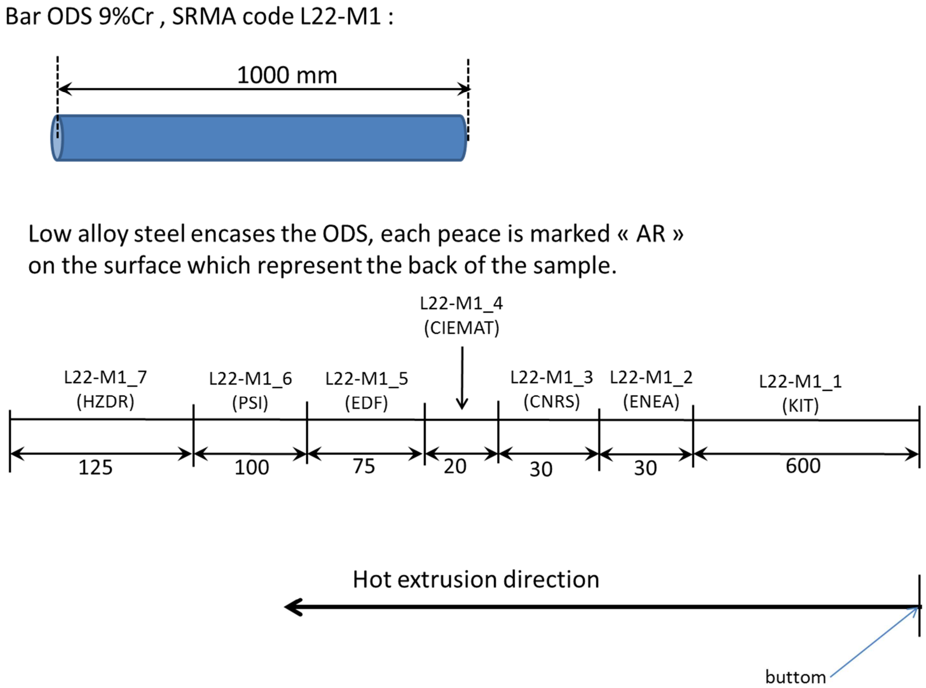

Figure 4.- Distribution of 9Cr-ODS Bar

4 ODS tubes

4.1 MATISSE 14Cr ODS Tube

Concerning the Fe-14Cr ODS tube (SRMA code : K48-T1) the intermediate heat treatments were performed at a temperature close to 1200°C followed by two cold rolling passes and an annealing at 750°C / 1h. The microstructure and mechanical properties should be close to Figures 5 [1] and 6, respectively.

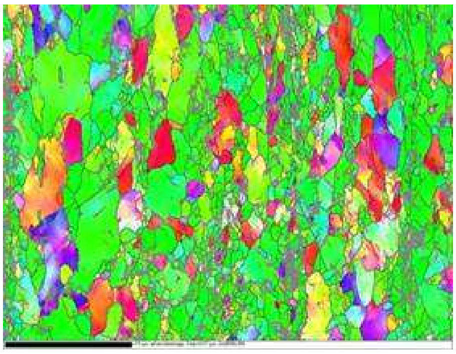

Figure 5.- Tensile properties of the 14Cr ODS tube

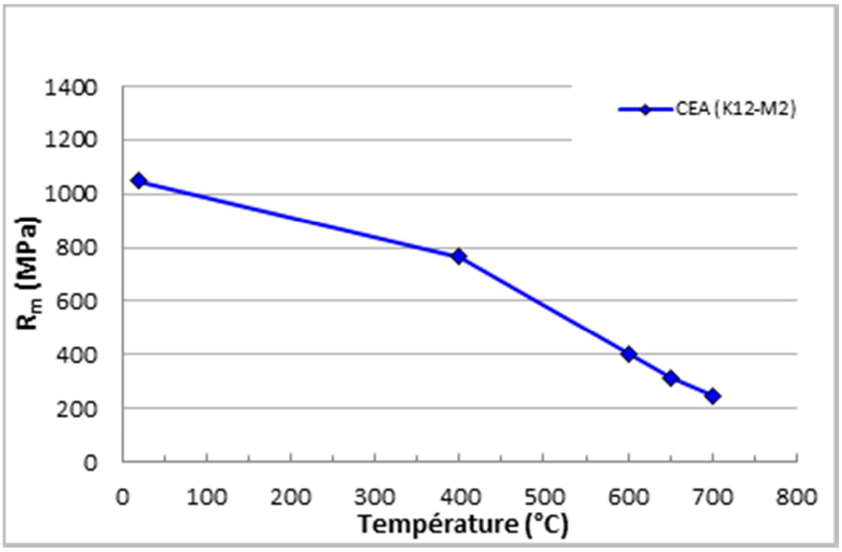

Figure 6.- Tensile properties of the 14Cr ODS tube

The distribution scheme of the 14Cr-ODS tube is summarized in Figure 7.

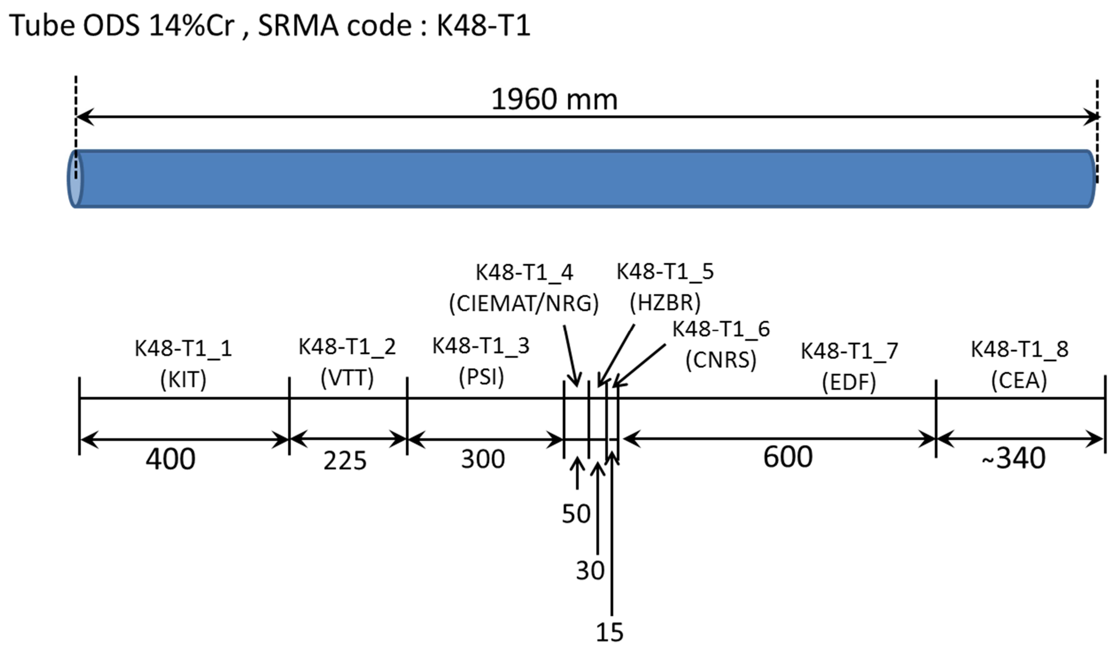

Figure 7.- Distribution of the 14Cr ODS tube

4.2 MATISSE 9Cr ODS Tube

Concerning the Fe-9Cr ODS tubes (SRMA code : K49-T1-M2 and K30-M3 both tubes are with the same fabrication route).

The intermediate heat treatments were performed at a temperature close to 1050°C, with a final normalization at 1050°C + annealing at 750°C. The microstructure and mechanical properties should be close to Figures 8 and 9, respectively.

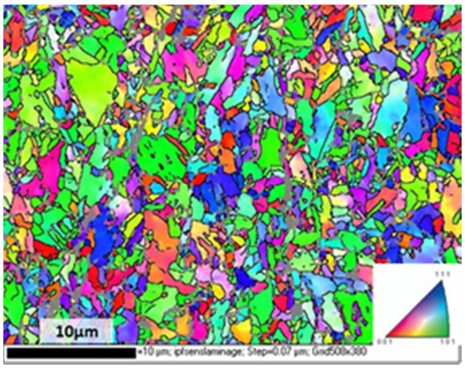

Figure 8.- EBSD map of the 9Cr ODS tube

And the mechanical properties, can be seen in the following figure

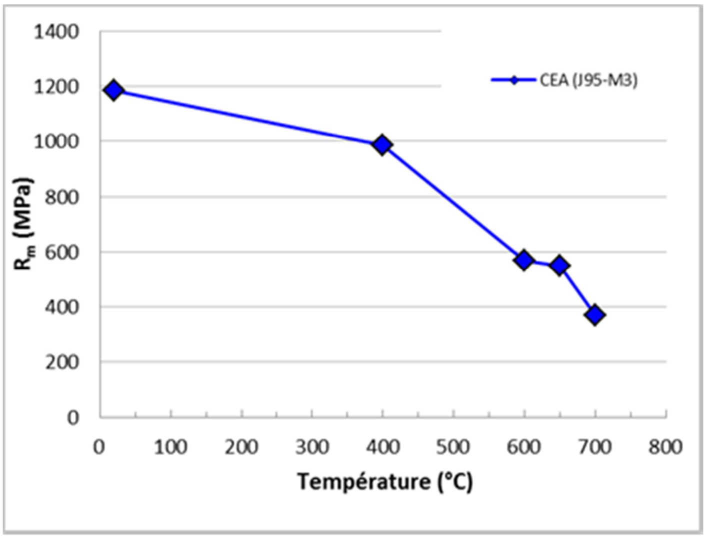

Figure 9.- Tensile properties of the 9Cr ODS tube

The distribution scheme of the 9Cr-ODS tube is seen in Figure 10.

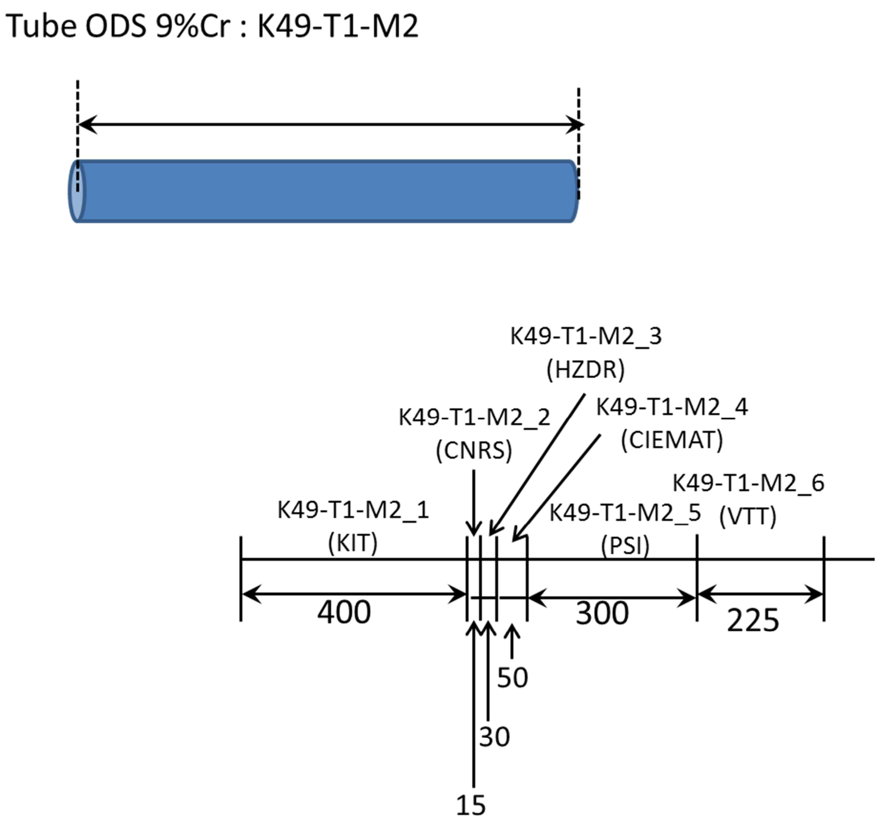

Figure 10.- Distribution of the 9Cr ODS Tube

4.3 MATISSE 18Cr ODS Tube

For the plugging material, that is a Fe-18Cr ODS tube (SRMA code J54-T1),the distribution scheme is shown in Fig. 11.It should exhibit a similar microstructure compared to the K38-T1 and should be more ductile compared to the K48-T1.

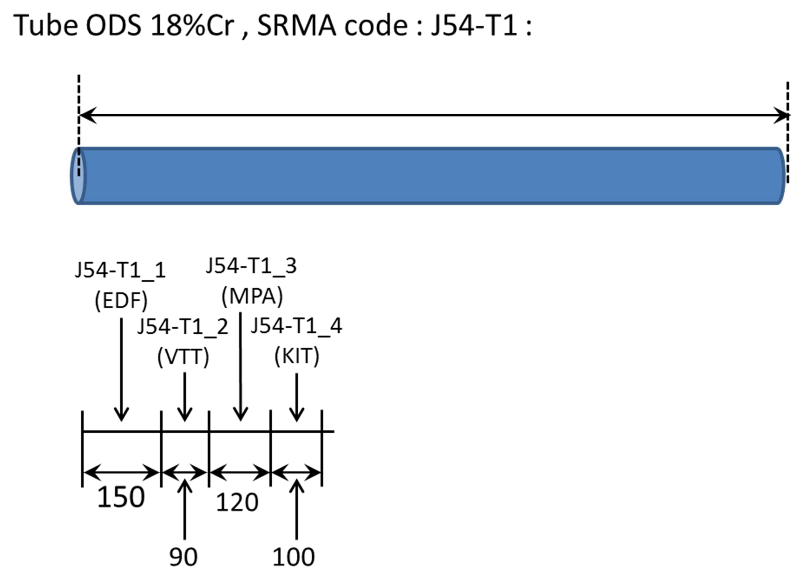

Figure 11.- Distribution of the 18Cr ODS tube

5 ODS plates

The GETMAT 12Cr-ODS and 9Cr-ODS plates were identified as suitable for the assessment of the GESA treatment on corrosion/oxidation behavior. Alfons Weisenburger (KIT-IHM) agreed to coordinate this activity. The plates are located at KIT and after the GESA treatment will be done, sample will be distributed to CIEMAT and CNR. Available cladding tubes will be distributed to PSI and SCK-CEN for the characterization under accident conditions. High temperature creep tests will be performed by CSM with available ODS bars.

5.1 GETMAT 12Cr-ODS plate

The 12Cr-ODS plate was produced by Kobelco within the GETMAT project with a pre-alloyed metal powder of composition (wt. %) 11.59Cr, 1.87W, 0.22Ti, 0.1Si and obtained by argon gas atomisation. The powders were mechanically alloyed in a dry type attrition ball mill with 0.23 wt. % Y_2O_3 and subsequently extruded at 1150ºC, hot forged at 1150ºC and annealed at 1150ºC for 1h. Finally, the forged plates were cold rolled with 40 % reduction and annealed for recrystallization at 1200ºC for 1 hour [2].

In all the sections studied of the 12Cr ODS plate it is possible to distinguish a bimodal grain size distribution, existing elongated small grains with sizes between 200 nm and 5 µm and elongated large grains with sizes up to 20 µm, as shown in Figure 12. The aspect of the grains could be an indication of an incomplete recrystallization during the fabrication process. EBSD maps show that the smallest grains seem to have an orientation along <110> in the extrusion direction, while larger grains have no preferential orientation.

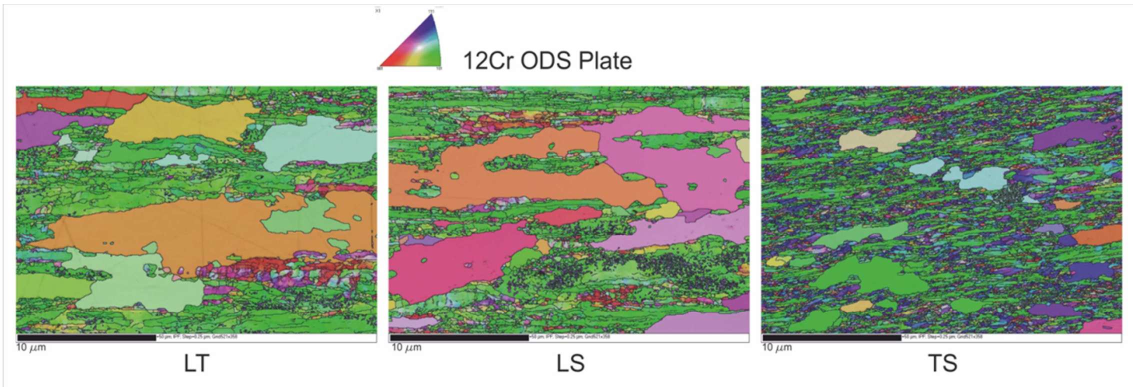

Figure 12.- EBSD maps of the 12Cr-ODS Plate

5.2 GETMAT 9Cr-ODS plate

The 9Cr-ODS plate was produced by Kobelco within the GETMAT project with T91 powders obtained by argon gas atomisation. The powders were mechanically alloyed in a dry type attrition ball mill with 0.23 wt. % Y2O3 and subsequently extruded at 1150ºC, hot forged at 1150ºC and annealed at 780ºC for 1h. Finally, the forged plates were cold rolled with 40 % reduction and final heat treated at 1050ºC for 1 hour and 750ºC for 1h [3].

Elongated grains are detected in both directions (perpendicular and in rolling direction). Most of these grains are around 20 µm large, occasionally grains up to 45 µm. Pores aligned in the rolling direction: average diameter between 1.7 and 4 µm, the large ones have mostly a size of about 5.3 to 5.5 µm.

6 Test matrix

6.1 Task 4.1. Microstructural examinations

A complete microstructure scenario of ODS alloys will be obtained by the combination of different techniques: TEM (EDF, KIT-IAM-AWP, CIEMAT), EBSD (EDF, KIT-IAM-AWP, CIEMAT), APT (CNRS), SANS (HZDR), n-diffraction (ENEA, CIEMAT), High-energy synchrotron diffraction, HESD (CIEMAT)

In addition thermal stability (EDF, CIEMAT, KIT-IAM-AWP) will be performed:

- Low temperature: 400 – 600 °C short term (approx. 1000 hours): Hardening Cr-rich α´ (CIEMAT)
- Intermediate temperature: 650 – 800 °C long term (up to 20.000 hours): Possible Softening – Large particles/grain size. (CIEMAT, EDF)
- High temperature (900ºC, 1100ºC) short term (up to 2500 h) (KIT-IAM-AWP)

Ion irradiation will be performed by (CEA, HZDR). The conditions will be defined later.

Deformation mechanisms will be studied by CEA, CIEMAT, HZDR, PSI, KIT using different techniques: In-situ TEM (CEA, CIEMAT), HESD (CIEMAT), Small punch tests (CIEMAT, HZDR), In-situ neutron diffraction (PSI), Cyclic deformation (KIT). A detailed table on task 4.1 activities is included in the Annex.

6.2 Task 4.2. Cladding tubes characterization

Microstructure characterization of the 9Cr ODS and 14Cr ODS cladding tubes include: TEM (EDF, CIEMAT), SEM/EBSD (EDF, CIEMAT), APT (CNRS), SANS (HZDR), and High-energy synchrotron diffraction, HESD (CIEMAT).

Some thermal stability analysis is also included (CEA, CIEMAT)

Mechanical characterization of the ODS steel tubes under uniaxial and biaxial loading will be performed to assess the impact of the microstructure anisotropy on the mechanical behavior, including

- Tensile tests: Uniaxial (CVR), Internal pressure (KIT, MPA),
- Creep: Small specimens (axial and hoop PSI), Biaxial (VTT), Internal pressure (EDF)
- Ring tests (INR)
- Segmented cone mandrel test (JRC-IET)
- Internal Conical Mandrel tests (CEA)

A detailed table on task 4.2 activities is included on the Annex.

6.3 Task 4.3: Characterization of ODS under safety-related operating conditions

The activities within this task are:

- Characterization of ODS tubes under off-normal conditions as high temperature/stress incursions (PSI)
- Creep tests at high temperature representing off-normal conditions (CSM)
- Burst tests (SCK CEN)
- ODS surface treatment (melting) with e-beams for improving the resistance against the action of hostile environments. Sample distribution (KIT) and oxidation corrosion tests (KIT, CIEMAT, CNR).

The tests matrix for corrosion experiments is shown in the following tables

Constant temperature:

	Temperature [°C]	Oxygen content [wt%]	Time [h]	remarks	samples
9Cr ODS	550, 600, 650 ,700	10^{-6}	2000,5000, 10000	CIEMAT, KIT	24, 24
12 Cr ODS	550, 600, 650 ,700	10^{-6}	2000,5000, 10000	CIEMAT, KIT	24, 24
9Cr ODS	450, 650	Red.	2000,5000, 10000	CNR	24
12 Cr ODS	450, 650	Red.	2000,5000, 10000	CNR	24

Temperature transients:

KIT (IHM) / 4 samples

	Temp. [°C]	O-content [wt%]	Time [h]	remarks
9Cr ODS	550	10^{-6}	5000	After 2000h to 750°C for 24h
12 Cr ODS	550	10^{-6}	5000	After 2000h to 750°C for 24h

CIEMAT / 4 samples:

	Temp. [°C]	O-content [wt%]	Time [h]	remarks
9Cr ODS	550	10^{-6}	5000	After 2000h to 1000°C for 24h
12 Cr ODS	550	10^{-6}	5000	After 2000h to 1000°C for 24h

CNR / 4 samples

	Temp. [°C]	O-content [wt%]	Time [h]	remarks
9Cr ODS	450	Ar+/ red	5000	After 2000h Ar+ →red
12 Cr ODS	450	Ar+/ red	5000	After 2000h Ar+ →red

7 References

1 R.E. Logé, L. Toualbi, E. Vanegas-Marques, Y. de Carlan, K. Mocellin, "Optimization of the fabrication route of ferritic/martensitic ODS cladding tubes: metallurgical approach and pilgering numerical modeling", IAEA International Conference on Fast Reactors and Related Fuel Cycles: Safe Technologies and Sustainable Scenarios (FR13), June 2013

2 Annette Heinzel, Alfons Weisenburger, Rainer Lindau, Georg Müller, "Production and characterization of a 9CrMoVNb ODS steel", GETMAT D1.3, Jun 2012

3 Annette Heinzel, Alfons Weisenburger, Rainer Lindau, Georg Müller, "Production and characterization of a 9CrMoVNb ODS steel", GETMAT D1.3, Jun 2012

ANNEX 1: Detailed tables

Table 1.- Material availability and characterization techniques

Material	Task 4.1		Task 4.2	Task 4.3
14Cr-ODS Bar (GETMAT)	TEM (EDF, KIT-IAM-AWP) EBSD (EDF, KIT-IAM-AWP) APT (CNRS) SANS (HZDR) N-diffraction (ENEA, CIEMAT) High-energy synchrotron diffraction, HESD (CIEMAT)	Nanoindentation (HZDR, CIEMAT) In-situ TEM (CEA, CIEMAT) Small punch (HZDR, CIEMAT) Thermal stability (EDF, CIEMAT) Ion irradiation (CEA, HZDR)		
9Cr-ODS Bar (MATISSE)	TEM (EDF, KIT-IAM-AWP) EBSD (EDF, KIT-IAM-AWP) APT (CNRS) SANS (HZDR) N-diffraction (ENEA, CIEMAT) High-energy synchrotron diffraction, HESD (CIEMAT)	Nanoindentation (HZDR, CIEMAT) In-situ TEM (CEA, CIEMAT) Small punch (HZDR, CIEMAT) Thermal stability (EDF, CIEMAT) Ion irradiation (CEA, HZDR) Cyclic behavior (KIT-IAM-WBM2) In situ neutron diffraction (PSI)		Creep at high temperature (CSM)

Table 1.- Material availability and characterization techniques (contd.)

Material	Task 4.1		Task 4.2	Task 4.3
18Cr-ODS cladding tube (MATISSE)			Pressurized tube plugging (EDF, VTT, MPA, KIT-IAM-WBM2)	Burst tests (SCK-CEN)
14Cr-ODS cladding tube (MATISSE)	TEM (EDF, KIT-IAM-AWP) EBSD (EDF, KIT-IAM-AWP) APT (CNRS) SANS (HZDR) N-diffraction (ENEA, CIEMAT) High-energy synchrotron diffraction, HESD (CIEMAT)	Thermal stability (EDF, CIEMAT) Small punch (CIEMAT)	Uniaxial tensile (CVR) Biaxial creep tests (EDF, VTT) Internal pressure tests (MPA, KIT-IAM-WBM2) Mandrel tests (CEA, JRC) Ring creep tests (PSI) Ring tests (INR)	Fracture behavior (PSI)
9Cr-ODS cladding tube (MATISSE)	TEM (EDF, KIT-IAM-AWP) EBSD (EDF, KIT-IAM-AWP) APT (CNRS) SANS (HZDR) N-diffraction (ENEA, CIEMAT) High-energy synchrotron diffraction, HESD (CIEMAT)	Thermal stability (EDF, CIEMAT) Small punch (CIEMAT)	Uniaxial tensile (CVR) Biaxial creep tests (EDF, VTT) Internal pressure tests (MPA, KIT-IAM-WBM2) Mandrel tests (CEA, JRC) Ring creep tests (PSI) Ring tests (INR)	
12Cr-ODS Plate (GETMAT)				GESA (KIT-IHM) Oxidation/Corrosion (KIT-IHM, CNR, CIEMAT)
9Cr-ODS Plate (GETMAT)				GESA (KIT-IHM) Oxidation/Corrosion (KIT-IHM, CNR, CIEMAT)

Table 2.- Task 4.1 activities on 9Cr-ODS Bar

Partner	Type of activities	Fe9Cr ODS As-received	Fe9Cr ODS Th. treatment	Fe9Cr ODS Post-anneal exam	Fe9Cr ODS Ion irradiation	Fe9Cr ODS Post-irrad. exam.
CEA	Ion irr., th. aging, μ-plasticity	μ-plasticity	Conditions to be defined	μ-plasticity	Conditions to be defined	μ-plasticity
CIEMAT	Th. stability, fracture, NRG part		700ºC/10.000h, 850ºC/10.000 h and 475ºC/1000 h	μ-plasticity		
CNRS	APT/TEM					
EDF	Chem. analysis, th. aging	Chemical analysis	600°C-650°C/15000h-20000h	who, what?		
ENEA	Neutron diffraction	Neutron diffraction		Neutron diffraction		
HZDR	SPT	SPT				
HZDR	Ion irradiation + PIE				Conditions to be defined	Nanoindentation/TEM?
HZDR	SANS + SEM/EDX + nanoind.	SANS, SEM/EDX, Nanoind.	-	-		
KIT	Mechanical properties	Mechanical properties				
KIT	Thermal aging		900°C,1100°C/100h,500h,2500h	TEM, EBSD		
PSI	In situ neutron diffraction					

Table 3.- Task 4.1 activities on 14Cr-ODS Bar

Partner	Type of activities	Fe14Cr ODS As-received	Fe14Cr ODS Th. treatment	Fe14Cr ODS Post-anneal exam	Fe14Cr ODS Ion irradiation	Fe14Cr ODS Post-irrad. exam.
CEA	Ion irr., th. aging, µ-plasticity	µ-plasticity	Conditions to be defined	µ-plasticity	Conditions to be defined	µ-plasticity
CIEMAT	Th. stability, fracture, NRG part		700ºC/10.000h, 850ºC/10.000 h and 475ºC/1000 h	µ-plasticity		
CNRS	APT/TEM					
EDF	Chem. analysis, th. aging					
ENEA	Neutron diffraction					
HZDR	SPT	-				
HZDR	Ion irradiation + PIE				Conditions to be defined	Nanoindentation/TEM?
HZDR	SANS + SEM/EDX + nanoind.	SANS, SEM/EDX, Nanoind.				
KIT	Mechanical properties					
KIT	Thermal aging					
PSI	In situ neutron diffraction					

Table 3.- Task 4.2 activities on 9Cr and 14Cr tubes

Partner name	Tests on 9Cr and 14Cr tubes
CEA	Material distribution Mandrel tests to determine DTT tensile
CNRS	APT
CIEMAT	Small punch tests, also thermal treatment, up to 550C SEM/EBSD, TEM, nanoindentation, neutron diffraction
CVR	tensile uniaxial
EDF	creep testing using internal pressure with and without ageing, SEM/EBSD, TEM
ENEA	neutron diffraction
HZDR	SANS
JRC	Cone mandrel test
	microstructural characterisation
KIT	internal pressure tests combined internal tests/tensile tests
MPA	internal pressure tensile tests
	internal pressure fatigue tests
PSI	ring creep tests
INR	ring tests
VTT	biaxial creep tests