MATISSE WP4 Summary of first 18 months

The main achievements during this 1st 18 months period are:

- Distribution of ODS bars and cladding tubes (CEA)
- Preparation and distribution of GESA-treated ODS plates (KIT)
- Definition of testing conditions (CEA, CIEMAT, HZDR, KIT, VTT)
- Microstructural examination of bars and tubes: initial results available (CEA, CIEMAT, EDF, HZDR, ENEA)
- Microplasticity of ODS alloys studies: preliminary results available (CEA, PSI)
- Fracture toughness and small punch results of ODS cladding tubes (CEA, CIEMAT)

Significant results

Task 4.1: Role of the microstructure on the mechanical behaviour

ODS bars of 14Cr (from GETMAT project) and 9Cr has been distributed to the participants. 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. The Fe9Cr ODS Bar (SRMA code : L22-M1) is an extruded bar in the ferritic state fabricated and distributed by CEA. In order to correctly simulate the behaviour 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.

Small punch tests have been performed on the as received bars (HZDR, CIEMAT) and after thermal ageing of 475ºC/1000 h (CIEMAT). The Small Punch transition temperature of the 14Cr ODS bar is around -125ºC, see Figure 4.1. The measured Small Punch transition temperatures and the values converted to the Charpy-based transition temperature T41J are summarized in Table X. For ODS Fe-14Cr, the value given in Table X compares reasonably well with the T41J values deduced from the KLST impact tests performed in GETMAT (35 °C and 165 °C for orientations AR and RA, respectively). Preliminary small punch tests in the 14Cr ODS and 9Cr ODS bars aged at 475ºC/1000 h show some hardening, more significant for the higher Cr content.

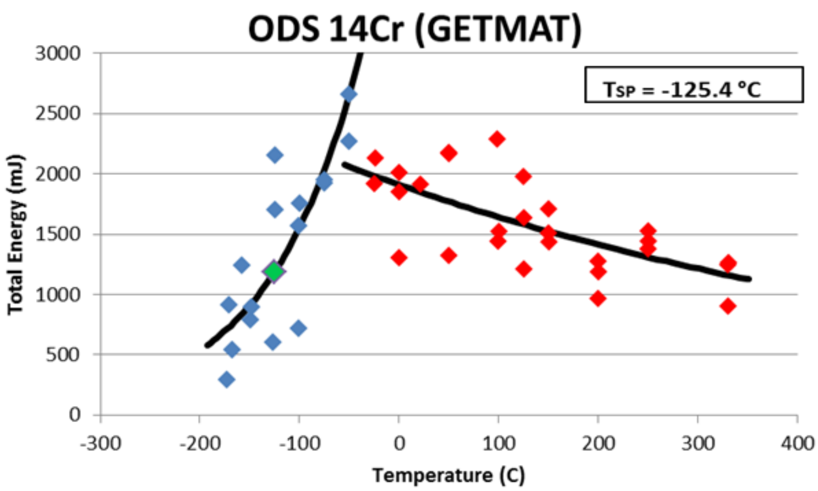


Fig. 4.1 – Transition temperature characterization of the 14Cr ODS bar by small punch tests (HZDR).

Different thermal ageing experiments are on going_

- 700ºC/10.000h and 800ºC/10.000h (CIEMAT)
- 900ºC and 110ºC for 100. 500 and 2.500 hours (KIT)
- 750ºC/15.000 hours (EDF)

In situ TEM straining experiments have been carried out at different temperatures by CEA on the 14Cr ODS Bar, see figure 4.2. The main points are the following: The hardening role of the precipitates is underlined at room temperature, with dislocations pinned on the nano-oxides. For high temperature, thermal activation enables the dislocations to get through the precipitates more easily. The deformation mechanism is modified with the temperature, with an intragranular character turning into intergranular for higher temperatures.

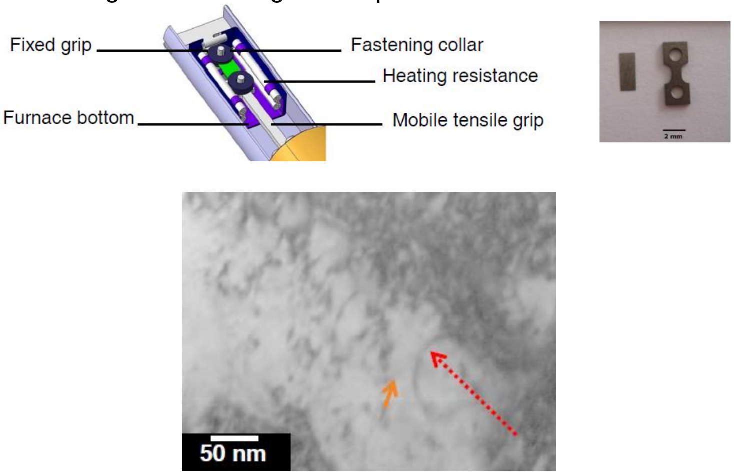


Fig. 4.2 – In-situ Tem deformation at different temperatures of 14Cr ODS (CEA)

In-situ neutron diffraction during tensile deformation of the GETMAT 9Cr ODS (provided by KIT), GETMAT 12Cr ODS (provided by KIT) and CEA 9Cr ODS Bar (CEA-SRMA code: L22-M1) were performed at PSI. The diffraction pattern, lattice strain during tensile test and the diffraction peak width changes during tensile test are under evaluation. The evolution of the lattice strains, for different planes perpendicular to the loading axis with applied load will gives information about the micromechanical behavior of the respective grain orientations parallel to the loading direction

Beam time proposal for Ion irradiation of 14Cr ODS and 9Cr ODS bars at HZDR were accepted. The conditions for the irradiation are: Fe-ions, successive irradiation at three ion energies of 0.5 MeV, 2 MeV and 5 MeV, two irradiation temperatures ofof 300 °C and 500 °C.

Task 4.2 - Characterization of ODS cladding tubes

14Cr ODS and 9Cr ODS cladding tubes were distributed by CEA. The distribution will be done in two steps. The first delivery prioritized those partners that are going to carry out creep experiments and thermal ageing treatments. A second delivery will be done in 2015 for the remaining partners. Around 1.8 meters of each tube (9Cr-ODS and 14Cr-ODS) were distributed by June 2014, and it is expected to have a similar quantity for the second step. In addition a 18Cr-ODS cladding tube were distributed for plugging trials of pressurized tubes. In Figure 4.3 the distribution of the tubes are shown.

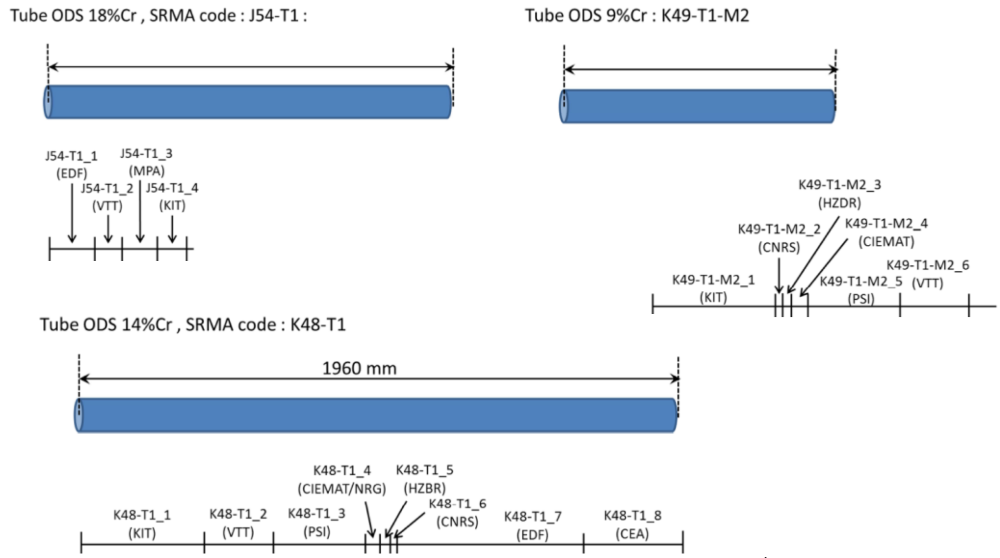
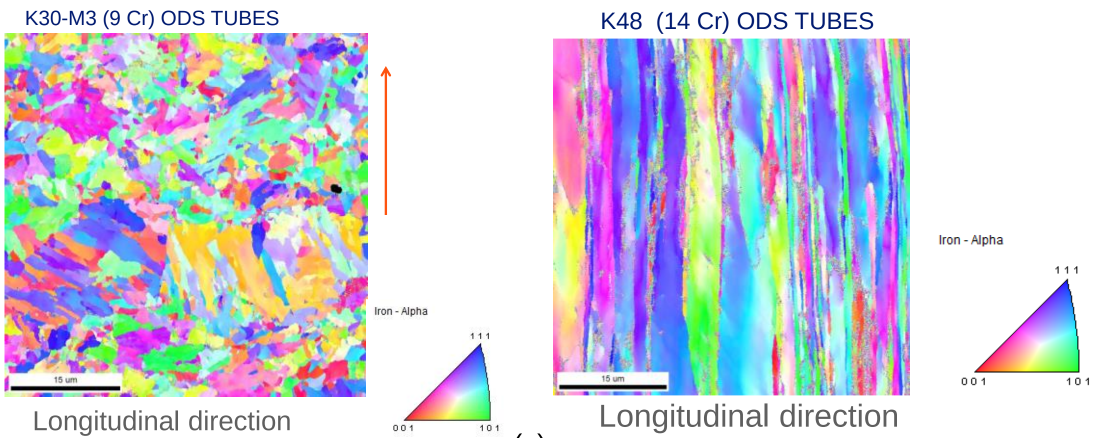


Fig. 4.3.- Distribution of ODS cladding tubes, 1st delivery

First microstructural examination of both tubes is available (CIEMAT, EDF). Figure 4.4 shows the EBSD images for the 9Cr and 14Cr ODS cladding tubes performed by EDF. Martensitic 9Cr ODS steels have predominantly equiaxed and homogeneous grain structure with a mean diameter of few µm. The EBSD map from longitudinal direction reveals that the grains are elongated, with a size ~10 in the longest dimension and a few hundreds nanometers in the transverse direction. The precipitates are chromium carbides (probably Cr23C6) with a size between tens and few hundreds nanometers. Titanium precipitates are also observed with a size of tens nm. This precipitates contains Y. The EDX confirm that the large precipitates are chromium carbides (probably $Cr_{23}C_6$). Yttrium are detected in the titanium precipitates with a size of tens nm. These precipitates are closed to the chromium carbides. The microstructure of the K48 (14Cr) ODS tube is textured and have an anisotropic grain morphology. The grains are elongated along the extrusion direction, with a size of tens micrometers in the longest dimension and a few micrometers in the transverse direction.

TEM examination of the tubes were discussed and it was agreed that CIEMAT will perform the general TEM examination and EDF will focused on the deep analysis of the nano-precipitates. The deliverable D4.21 Microstructure of ODS tubes will be issued with the available data (EBSD and TEM) and a second revision of the deliverable will be done as soon as all the microstructural analysis (TEM, APT, SANS) is finished. The SANS beam-time proposal was accepted by ILL Grenoble, the SANS experiment will take place on 23rd of June, 2015



(a) (b)

Fig. 4.5.- EBSD maps of the 9Cr (a) and 14Cr (b) ODS cladding tubes (EDF)

Small punch of the 9Cr and 14Cr ODS cladding tubes were performed at CIEMAT. Specimens of 3 mm diameter and 0.250 mm thickness were mechanized from the tubes wall. The small punch tests were performed at room temperature. The small punch behavior of the tubes, with samples oriented in the T direction are in between the behavior of the bars in both orientation (L and T), see Figure 4.6. That means that tube hoop behavior is improved, compared to the behavior of the hoop direction of the former bar. On the other hand, a slight better behavior is observed for the 14Cr ODS tube, compared to the 9Cr ODS tube.

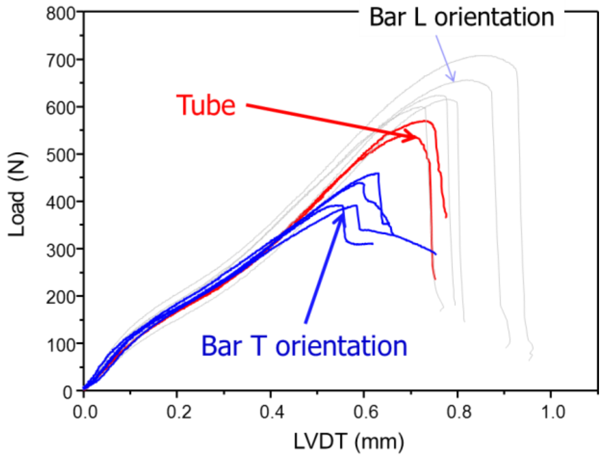


Fig. 4.6.- Small punch tests of 14Cr ODS cladding tubes. Comparison with tests performed in the 14Cr ODS bar in two orientations (CIEMAT)

Fracture toughness tests on ODS cladding tubes were performed by CEA by using Internal Conical Mandrel tests plus finite element simulation. In general there is no so much difference between the fracture toughness data regarding the chemical composition of the tubes (9Cr and 14Cr). Toughness (K0,2) are between 80 et 180 MPa.m1/2 that is superior to a previous Ramsès criteria (20MPa.m1/2).

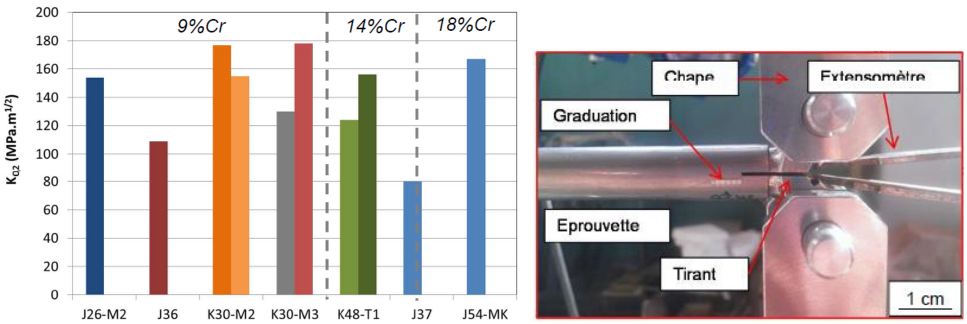


Fig. 4.7. Fracture toughness tests on ODS cladding tubes (CEA)

Tube plugging trials for pressurized tube testing are ongoing to fabricate the specimens for creep tests of cladding tubes (VTT, EDF, MPA, KIT). Some mechanical plugging systems are considered, but the technique is not already improved.

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

Electron-beams surface treatments for improving the coolant compatibility were already performed on selected 9Cr ODS and 12Cr ODS GETMAT plates (KIT). Oxidation and corrosion testing under relevant FNR coolants (Pb) at high temperature will be performed by KIT, CIEMAT and CNR. The material was already distributed. Corrosion tests of the same material in liquid Pb performed within GETMAT was summarized. SSRT of surface treated ODS will be done at SKC CEN.