EFDA WORKPROGRAMME 2010

Call for Participation

Emerging Technologies

Fusion Materials Topical Group

Research Project:
MAT-ODSFS: Nano-structured ODS Ferritic Steel Development

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1. Introduction

At its meeting in Barcelona on the 8th July 2009 the EFDA Steering Committee approved the EFDA 2010 Workprogramme, including the tasks identified below. This Call for Participation covers the activities of the 2010-WP of the MAT-ODSFS Research Project of the Fusion Materials Topical Group, Nano-structured ODS Ferritic Steel Development, and will be implemented on the basis of the provisions given in Art. 5 of the EFDA Agreement.

2. Objectives

The plate supporting the W tiles of the dual-coolant Tritium breeding blanket, and the cooling system for the He-cooled divertor are presently the foreseen applications of ODS Ferritic Steels (ODSFS). Both require materials that have sufficient creep strength in the temperature range of up to ~750°C, as well as reasonable fracture toughness.

The MAT-ODSFS Research Project on Nano-structured ODS ferritic steels development for DEMO has been organised along the following programmatic lines:

- Developing the present generation of nano-structured ODSFS.
- Industrial fabrication of the present generation of nano-structured ODSFS.
- Developing an optimised generation of nano-structured & nano-grained ODSFS.
- Stability of present and optimised generation of nano-structured ODSFS under creep and irradiation.

The main objectives of the WP2010 programme are summarised as follows:

(i) Developing the present generation of nano-structured ODSFS

The objective of developing the present generation of nano-structured ODSFS consists in optimising the chemical composition and determining a set of parameters for the Mechanical Alloying (MA) process and the subsequent Thermal-Mechanical Treatments (TMT), aimed at reproducing the main microstructure features identified as responsible for the high strength and the limited loss of fracture toughness after irradiation.

The optimisation will concentrate on chemical composition of the 14Cr-Y-W-Ti (14YWT) type, and complement the activities of the 2008-2009 by considering atom percent Ti to atom percent Y ratios higher than 1, highly energetic MA and hot extrusion at rather low temperature, down to ~850°C, to obtain a high density of nano-clusters enriched in Ti-Y-O, and high yield strengths of 800 and 450 MPa, respectively at Room Temperature (RT) and 600°C, associated with DBTT comparable to that of Eurofer.

Limited studies of microstructure under ion-beam irradiation (dpa & He production), addressing ODS stability, He accumulation in bulk and grain-boundaries, will have also to be carried out on the materials expected to be obtained with the most promising route.

(ii) Industrial fabrication of the present generation of nano-structured ODSFS

Following the withdrawal of Plansee from manufacturing ODSFS, there is no longer an industrial company in the EU capable of manufacturing the required range of ODSFS in a sustainable way. The situation could change once the decision on the future build of Generation IV sodium-cooled fast reactor has been made. This can potentially provide sufficient incentive to manufacturers to re-enter the ODS steels market. In the meantime we follow the two lines of development of ODS steels outlines below.

Based on the optimisation of the fabrication procedure (WP-08-09-10) and on the assessment of the possibility of fabricating, at a semi-industrial scale, a ~10 kg batch of a nano-structured ODSFS of the present generation via different sub-contractors coordinated by an Association (WP-08-09), an order will be placed, with a delivery date mid 2011.

In parallel, EFDA, in close collaboration with the Associations and F4E, will **examine the new Japanese offer of fusion relevant ODSFS by Kobe steel expected to be proposed during ICFRM-14** under the umbrella of the Implementing Agreement (IA) of the IEA.

Material production and specimens machining of 14-18%Cr ODS ferritic steels, and the subsequent study of microscopic processes associated with cleavage (transgranular) fracture: Charpy testing of thermally aged materials, fractographic and microstructural examinations will be carried out.

(iii) Developing an optimised generation of nano-structured & nano-grained ODSFS

ODS ferritic steels as driven system

The 2010 Work Programme will be devoted to the continuation and the completion of the work of 2009 in the framework of the Reactive Milling approach: (i) influence of the intensity, temperature and duration of milling, on the nano-cluster formation, (ii) consequences of the initial selection of the reactants on the nano-cluster formation and oxygen content in the matrix, (iii) effects of the annealing conditions on the nano-clusters characteristics. This will result in the definition of the first optimised fabrication route of 14YWT ODSFS using Reactive Milling.

ODS ferritic steels with improved plasticity.

The nano-grain sized 14YTW alloys will be produced on the laboratory scale using (i) High Energy Ball Milling (HEBM), (ii) consolidation, i. e. extrusion and/or spark plasma sintering, and (iii) thermo-mechanical treatment (TMT), the latter allowing high deformation rates under cold, warm (~400°C) or hot (~1000°C) processing conditions such as High Speed Hot Extrusion (HSHE) and Equal Channel Angular Pressing (ECAP). The effect of hot extrusion parameters on microstructure and mechanical behaviour will be characterised to select the best fabrication route for an optimised strength-ductility relation. The microstructure is expected to be characterized using transmission electron microscopy (TEM), atom probe tomography (APT), and related methods.

Optimised Generation of nano-structured & nano-grained ODSFS

Provided that the effort in WP-2008-2009 is sustained, the achievement of both objectives outlined above should provide data for the assessment of feasibility of producing optimised nano-structured and nano-grained ODSFS in the years 2012-2013, with the first specification ready by the end of 2012, and fabrication performed early in 2013.

(iv) Stability of present and optimised generation of nano-structured ODSFS under creep and irradiation

This line of research, starting within this Work-Programme 2010, will concern the characterisation of:

(i) Both lab scale batches produced in 2009.
(ii) Semi-industrial scale batch produced by mid-2011.
(iii) MA produced ODSFS of 14YTW ODSFS type for fission application.
(iv) Alternatively produced ODS martensitic steels for fission application.

The programme will focus on:

- Stability of the nano-clusters under dpa, He and H accumulation, using ion-beam irradiation and characterisation by TEM complemented by APT for the chemistry of the clusters.
- Creep resistance & fracture toughness or DBTT to assess the trade-off between both properties

In relation to the ODS steels developed for fission applications, the present work programme will complement the effort in the fission programmes, and will focus on the effect of He and H accumulation on the microstructure and stability of oxide nano-clusters.

On this basis, the most promising steel types, the desirable range of irradiation conditions in terms of spectrum, dose & temperature, and the type of Post-irradiation Testing will be defined in 2012

3. Work Description and Breakdown

3.1 Work Breakdown

Within the structure and the objectives defined above the Work Breakdown will be as follows.

(i) Developing the present generation of nano-structured ODSFS

The optimisation will concentrate on 14YWT chemical composition with high atom percent Ti to atom percent Y ratio, highly energetic MA and hot extrusion at rather low temperature to obtained a high density of nano-clusters enriched in Ti-Y-O, high yield strengths of 800 and 450 MPa, respectively at Room Temperature (RT) and 600°C, associated with DBTT remaining comparable to that of Eurofer. Specifically, the activities within 2010 will aim at:

(i) Complementing the optimisation of the powder metallurgy process (MA versus milling, with atom percent Ti to atom percent Y ratio higher than 1, pre-alloyed versus elemental powder, H versus Argon atmosphere, control of oxygen & degassing procedure).
(ii) Optimising the thermal-mechanical treatment (HIP, Hot extrusion, Hot and warm rolling…) versus microstructure, crystallographic texture and short term response mechanical properties (tensile and Charpy).

Small-Angle Neutron Scattering (SANS) and APT are mandatory in addition to TEM for assessing the nano-cluster dispersion.

Limited studies of microstructure under ion-beam irradiation (dpa & He production), addressing the microstructural stability of ODS, He accumulation in bulk and grain-boundaries, also will be carried out using the most promising materials identified as a result of the execution of the tasks.

The deliverables are summarised in the table below:

2010-WP: Developing the present generation of nano-structured ODSFS
Deliverables
03/2010: Report: *specification of the EFDA programme of characterization of the laboratory scale (1kg) batches*
12/2010 Interim Report on Laboratory scale batches: *First results on characterization: microstructure (TEM, APT, SANS), texture, tensile and Charpy with fractographic examination.*
12/2010: Interim Report on: Present generation of ODSFS*: Optimised chemical composition, MA and TMT versus microstructure (TEM, APT, SANS) & short term response mechanical properties, and, elements of radiation stability.*
12/2010: Report on: *14-18%Cr fission ODS ferritic steel microstructural examinations of 14-18%Cr ODS ferritic steel, Charpy, tensile and fatigue tests****,*** *microstructural and fractographical examinations of 14%Cr ODS ferritic steel up to high temperature*

(ii) Industrial fabrication of the present generation of nano-structured ODSFS

Based on the experience accumulated within the Work-Programme 2008-2009, concerning the optimisation of the fabrication route & the assessment of the possibility of fabricating, at a semi-industrial scale, a ~10 kg batch of a nano-structured ODSFS of the present generation via different sub-contractors coordinated by an Association, an order will be placed, with a delivery date mid 2011.

In parallel, the Japanese proposal to be discussed at ICFRM-14 in September 2009, will be assessed. In case of positive assessment an EFDA characterisation programme will be specified.

The deliverable is:

2010-WP: Industrial fabrication of the present generation of nano-structured ODSFS
Deliverables
06/2010: Joint report*: Specification of EFDA programme of characterization of the Japanese material*
12/2010: Issue of Specification for ordering *the ~10 kg EU batch of nano-structured ODSFS to be ordered via coordinating different sub-contractors. Delivery date mid 2011.*
12/2010: Interim Report on*: the first results on the Japanese ODSFS: microstructure, tensile and Charpy tests and fractography examination.*

(iii) Developing the optimised generation of ODS nano-structured & nano-grained ferritic steels

ODS ferritic steels as driven system under MA and subsequent thermal-mechanical treatment

The 2010 Work Programme will be devoted to the continuation and the completion of the work of 2009 on the 14YTW composition: (i) influence of the intensity, temperature and duration of milling, on the nano-cluster formation, (ii) consequences of the initial selection of the reactants on the nano-cluster formation and oxygen content in the matrix, (iii) effects of the annealing conditions on the nano-clusters characteristics. This will result in the definition of a first optimised fabrication route of 14YWT ODSFS using Reactive Milling.

The main deliverables are:

2010-WP:Developing the optimised generation of ODS nano-structured & nano-grained ferritic steels
ODS ferritic steels as driven system under MA and subsequent thermal-mechanical treatment
Deliverables
12/10: Report: *The best kinetic pathway for reactive MA of 14YTW in terms of milling intensity & temperature, and annealing condition for a nano-structured ODSFS of 14YTW type, based on microstructure examination (APT, SANS). Elements for selecting the best initial reactants in the case of Reactive Milling*

Developing radiation resistant ODS ferritic steels with improved plasticity.

Nano-grained size 14YTW alloys will be produced and characterised at the laboratory scale using MA and various techniques of (i) High Energy Ball Milling (HEBM). (ii) consolidation, i. e. extrusion and/or spark plasma sintering, of (iii) TMT allowing high deformation rate under warm or hot process such High Speed Hot Extrusion (HSHE) an Equal Channel Angular Pressing (ECAP). These techniques will be used to achieve the best strength-ductility relation.

In addition to MET, Microstructure characterisation of the nano-clusters shall include Atom Probe Tomography (APT) to estimate their chemical composition and the content of oxygen in the matrix. A particularly important issue is the stability of the nano-clusters during the thermal mechanical treatments.

Heavy ion-beam irradiation will be used to have preliminary results on the radiation resistance of the obtained microstructure & nano-clusters in comparison with normal grain-sized steel of same chemical composition and similar distribution of nano-sized clusters.

The deliverables are:

2010-WP:Developing the optimised generation of ODS nano-structured & nano-grained ferritic steels
Developing radiation resistant ODS ferritic steels with improved plasticity
Deliverables
12/10: Report on*: the effect of selected parameters of consolidation High Speed and ECAP processes for ODS powders prepared by HEBM*
12/10: Report on*: production of nano-grain sized model 14YTW ODSFS. Determination of their microstructure and mechanical properties versus temperature*

(iv) Stability of ODS ferritic steels under creep and irradiation

The work-programme will rely on the 2009 report coordinated by EFDA defining this characterisation programme, issued 12/2009 (see WP08-09-MAT-ODSFS 13 November 2008).

The programme will focus on:

- Stability of the microstructure, especially the nano-clusters under dpa, He and H accumulation, using ion-beam irradiation and characterisation by TEM complemented by APT for the chemistry of the clusters.
- Creep resistance and the associated fracture toughness or DBTT to assess the trade-off between both properties

The deliverable is:

2010-WP: Stability of ODS ferritic steels under creep and irradiation
Deliverables
12/2010: Interim Report on: the Japanese material *First results of characterization.*
12/2010: Report on: Laboratory scale MA ODSFS batches: *Tensile & Fracture toughness, and stability under dual beam.*
12/2010: Report on: MA produced ODSFS for fission applications*: Stability under dual ion-beam irradiation assessed via TEM and APT*
12/2010: Report on: Alternatively produced ODSFS*: Stability under dual ion-beam irradiation assessed via TEM and APT*
12/2010: Interim report on: *First Creep tests on lab-scale and Japanese ODSFS.*

(v) Priority Support:

- The 2010 Work-Programme described above is proposed under Baseline Support, except the fabrication of the semi-industrial batch ~10 kg of 14YWT type nano-structured ODSFS via sub-contracting to industrial companies specialised in the various fabrication processes, which is proposed under Priority Support for 0.5 PPY and 60 k€, for powder procurement, Mechanical Alloying (MA) and TMT sub-contracting.

Projects	Manpower PPY	Manpwoer k€	Other Expenditure k€	Comments
Semi-industrial Fabrication a 10 kg batch of present generation	0.5		60	
Ion-beam time for radition stabilitry under single and dual beam irradiation			120	* 120k€ for beam time at JANNUS (20 days Orsay, 20 days Saclay). * 60 k€ ODSFS fabrication sub-contract & 0.5PPy for follow-up.
Sub-Total	**0.5**		**180**	

3.2 JET related activities

Non-applicable.

3.3 Publications

A list of publications produced on the basis of results of the 2010-WP, will be compiled after the completion of these tasks.

4. Scientific and Technical Reports

4.1 Progress reports

At the end of each calendar year and at intermediate times where appropriate, the Task Coordinator shall submit a report on activities under the Task Agreement to the EFDA Leader for his approval. These reports shall describe the progress made by each Association on each activity, and they shall indicate the level of achievement of the objectives, the status of the activities, the allocation of resources and recommendations for the next year where applicable. The EURATOM financial contribution will be made through the usual procedures for baseline support through the Contract of Association.

4.2 Report of achievements under Priority Support (final report and, when appropriate, intermediate reports):

The progress of tasks undertaken under Priority Support and the status of deliverables will be reported separately to the EFDA Leader. A final report (and intermediate reports indicating any substantial progress in the achievement of deliverables, if requested by the EFDA Leader) shall be prepared by the Task Coordinator and submitted to the EFDA Leader. These reports shall include specific sub-sections for each of the Associations involved. They shall document the degree to which the deliverables outlined have been achieved, and shall include a breakdown of expenditure for each Association. The EURATOM financial contribution will be made after approval by the EFDA Leader of these reports.

4.3 Milestones.

The results obtained within the Research Project Nano-structured ODS Ferritic Steel Development will be presented by the principal investigators and reviewed during joint monitoring meetings held twice a year. On this basis the progress accomplished by the contributing Associations will be reported by the Coordinator to the EFDA Leader.

The report on the Association activities under Priority Support will be prepared by the Coordinator to be presented to the EFDA Leader at the end of every calendar year.

The final and technical report will be submitted to the Responsible Officer of the Topical Group for approval and uploading the IDM database.

5. Association Proposal

The Associations are requested to complete the Association Response via ECoM system.