Agenzia Nazionale per le Nuove Tecnologie,
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RICERCA DI SISTEMA ELETTRICO

Realizzazione sezione di prova e provini per caratterizzazione fuori pila di materiali strutturali

A. Gessi

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Report RdS/2010/104

REALIZZAZIONE SEZIONE DI PROVA E PROVINI PER CARATTERIZZAZIONE FUORI PILA DI MATERIALI STRUTTURALI

A. Gessi ENEA

Settembre 2010

Report Ricerca di Sistema Elettrico
Accordo di Programma Ministero dello Sviluppo Economico – ENEA
Area: Produzione e fonti energetiche
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Titolo

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Descrittori

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Argomenti trattati: Reattori Nucleari Veloci
Generation IV Reactors
Caratterizzazione dei materiali
Effetti delle radiazioni sui materiali

Sommario

La realizzazione di prove meccaniche (creep e tensile) di materiali innovativi, quali 9Cr ODs e 14Cr ODS, è in programmazione presso il Laboratorio Prove Meccaniche di Brasimone. Queste prove vengono condotte parallelamente alla attività LEXURII, parte del programma europeo GETMAT, dove campioni dello stesso tipo vengono sottoposti alle medesime prove dopo un irraggiamento da spettro veloce, fino a 16dpa, a contatto con Pb, fino a 550°C. la conduzione di queste prove rende necessario l'aggiornamento delle strumentazioni del Laboratorio Prove Meccaniche al Brasimone.

Note

Il rapporto è emesso in Inglese.

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1. Mechanical testing-out of pile

1.1. Introduction

The need for an Out of Pile experimental campaign on ODS steels and reference materials goes in parallel with an importan irradiation experiment calle LEXURII. This experiment, where ENEA is task responsible, is part of the GETMAT FPVII program. ENEA will work jointly with SCK-CEN in order to perform a full irradiation experiment in the BOR60 reactor, at RIAR, Dimitrovgrad, RU. ENEA, in the frame of the National AdP, will carry on a comprehensive carachterization of the same materials, without irradiation, with the target to compare with the final results of the irradiation experiment. In LEXURII, ENEA will carry on the Pb exposure ; in this program ENEA pland to carry on both LBE and Pb tests, at 350°C and 550°C.

1.2. Specimens type

This experiment will test: 9cr ODS steel, 14Cr ODS steel, 12Cr ODS steel, 15-15Ti stabilized SS steel, T91 ferritic martensitic, T91 coated specimens.
The specimens kinds will be : Tensile, C(T), corrosion, pressurized tubes, coated specimens.

The tensile specimens will undergo mechanical tests in air, LBE and in molten lead at 350°C and 550°C respectively. The fractured surfaces will be analyzed by scanning electron microscopy (SEM) to determine the fracture mechanism. The tubular gas-filled specimens will be subject to measurements of dimensional changes to evaluate the irradiation creep properties of the different steels. The cross section of corrosion specimens will be analyzed by different techniques including optical microscopy, SEM and electron probe. The maximum amount of analysis is 42 for mechanical tensile tests, 24 for fractography, 24 for tubular gas-filled specimens and 24 for corrosion analysis.

2. Test matrix

2.1. Corrosion specimens

The objective of corrosion study is to evaluate the corrosion along a cross section. Corrosion layer thickness needs to be measured in few points on those cross sections. To perform the work, specimen will be embedded and properly polished. In addition a chemical composition along a line starting from the bulk to the corroded surface should be established. Element of importance are Fe, Cr, Ni, Pb, Bi, O and Al, Y for coated specimens. The procedure of preparation specimens for OM, SEM and EPMA is still to develop and agree between SCK•CEN/ENEA and RIAR. The aims of the procedure are (1) to avoid interaction between embedding resin and LBE and (2) prevent smearing of Pb/Bi on the cross section during preparation of the specimens.

2.2. Optical microscopy

Microstructure parameters. The output consists of a systematic set of photographs that characterize the microstructure features of the corrosion layer and subsurface layer. A macro photograph should be provided as an overview of the complete cross section. A set of photographs providing a higher magnification band across the cross section at 3 positions (minimum, maximum and average thickness of the corrosion layer).
The set of photographs of the corrosion specimens cross section is used to quantify the oxide layer thickness.

2.3. SEM

Analysis of the microstructure at 3 positions for every corrosion specimens cross section.
The set of SEM photographs at different magnifications (generally 800X, 2000X and 5000X) show a more detailed view on the corrosion and subsurface layers microstructure and same positions used in the optical microscopy. Back Scattered Electron (BSE) microscopy should be also performed to analysis of different phases distribution. Simultaneous recording of Secondary Electron and BSE to be provided for each image.

2.4. EPMA

Fe, Cr, Pb, Bi, O repartition for ferritic/martensitic steels
Fe, Cr, Ni, Pb, Bi, O repartition for austenitic stainless steels
Fe, Cr, Pb, Bi, O, Y repartition for ODS T91 steels
Fe, Cr, Ni, Pb, Bi, O, Al, Y repartition for coated specimens
Local line scans (3 selected positions). The scans are taken at the same positions used in the SEM. The output consists of a set of the elements distributions along the line from external surface of corrosion layer towards the

bulk of material up to the depth where bulk concentrations of major alloying elements is reached.

Other elements repartition necessary to understand mechanisms of corrosion. If the need arise, the subject will be discussed and justified in the technical meetings.

2.5. Dimension analysis of pressurized tubes

After irradiation, tubes are to be cleaned with a recipe that will be provided by ENEA/SCK•CEN. Dimensional measurements with accuracy no worse then +-3µm and visual examination (surface defects, scratches) should be performed before filling, after filling and after irradiation. Measurement at 3 locations (mid, left and right) is foreseen. For each position measurement at 0° and 90° should be performed.

2.6. Tensile tests

In order to ensure the reliability of the mechanical tests results the cross check of the tensile tests procedure should be performed prior to the irradiation specimens tests. The specimens of the unirradiated material (T91) fabricated at SCK•CEN will be tested independently at ENEA. The tests conditions are the following:

- in air at 350°C and at 550°C
- in LBE at 350°C
- in molten lead at 550°C.

The reasonable agreement between the results obtained at different labs should be reached. On this grounds SCK•CEN and ENEA will then make a decision to proceed with the tests of irradiated specimens.

In order to evaluate influence of irradiation alone one tensile specimens of every material will be cleaned from residuals of lead and LBE with a recipe that will be provided by ENEA/SCK•CEN and then test in the air. The test temperatures for these tests will be equivalent to the corresponding irradiation temperatures.
Other tensile specimens will be performed in the environment corresponding to the environment in which the specimens were irradiated. The exact amount of specimens of every material to test will be specified by SCK•CEN and ENEA later after performing of unirradiated material tests. However the total amount should not exceed 42 specimens. Environment for testing will be Pb or PbBi provided by ENEA batch of available material. Tests will probably be performed with a Pb/PbBi free surface in contact with air, therefore some getter will be placed (pure Mg or other appropriate material in solid form).

The strain rate for the test program will be 5 10E-6 or higher.

The exact specification of the test should be still agreed between SCK•CEN, ENEA and RIAR. The ASTM E8M-04 and ASTM E21-05 should be served as an example.

2.7. Fractography

The objective of the fractography work is to identify the fracture mode and initiation points. The idea is to better understand the fracture mechanism.

3. Conclusion