G Model
CS-6637; No. of Pages 10
ARTICLE IN PRESS
Corrosion Science xxx (2016) xxx–xxx
Contents lists available at ScienceDirect
Corrosion Science
journal homepage: www.elsevier.com/locate/corsci

The reproducibility of corrosion testing in supercritical water—Results of an international interlaboratory comparison exercise

D. Guzonas [a,*], S. Penttilä [b], W. Cook [c], W. Zheng [d], R. Novotny [e], A. Sáez-Maderuelo [f], J. Kaneda [g]

[a] Canadian Nuclear Laboratories, Chalk River Laboratories, Chalk River, ON K0J 1J0, Canada
[b] VTT Technical Research Centre of Finland Ltd., Nuclear Safety, P.O. Box 1000, Espoo FIN-02044 VTT, Finland
[c] University of New Brunswick, Department of Chemical Engineering, P.O. Box 4400, Fredericton, NB E3B 5A3, Canada,
[d] CanmetMATERIALS (CMAT), 183 Longwood Rd. S., Hamilton, ON L8 P 0A1, Canada
[e] JRC-IET, Westerduinveg 3, 1755 LE Petten, The Netherlands
[f] Structural Material Division (CIEMAT), Avda. Complutense, 40, Madrid 28040 , Spain
[g] Plant and Systems Engineering Section, Nuclear Plant Engineering Department Hitachi-GE Nuclear Energy, Ltd., United States

ARTICLE INFO

Article history:
Received 7 July 2015
Received in revised form 29 January 2016
Accepted 31 January 2016
Available online xxx

Keywords:
Steel
Weight loss
Oxidation
High temperature corrosion
SEM

ABSTRACT

A major challenge in supercritical water-cooled reactor development is the lack of a consistent alloy database. An international interlaboratory comparison test was organized to study the reproducibility of weight change data obtained for identical alloys under similar conditions in different facilities. This paper presents the test procedures, conditions, results, and additional characterization data. More variation in weight change was observed than expected. The scatter was small within the same laboratory, but large between different laboratories. Much of this variation appears to be attributable to differences in test facilities. The data generally agree on the relative ranking of the corrosion resistance.

1. Introduction

The use of supercritical water (SCW) as the coolant in a nuclear reactor is the logical evolution of the current generation of water-cooled reactors, which generate almost all of the electricity produced by nuclear power worldwide. The use of SCW as the coolant in nuclear reactors increases the efficiency over that of currently operating nuclear power plants, decreases capital and operational costs, and decreases electrical energy costs. Water is a familiar and relatively safe heat transfer medium, and many power utilities already operate both nuclear power plants and fossil-fired SCW power plants (FFSPs), and can easily see the technical synergies.

Selection of materials for the fuel cladding and other in-core components for a supercritical water-cooled reactor (SCWR) is a key challenge in concept development. While zirconium alloys have a low neutron capture cross-section and remain the preferred fuel cladding alloy choice from the perspective of neutron economy, it has been long known that these alloys experience unacceptably high corrosion rates in SCW [1]. Although some Zr–Fe–Cr alloys showed promise at 500 °C, it has been shown [2,3] that these alloys can also experience breakaway corrosion at that temperature. In addition, zirconium alloys typically have poor high-temperature mechanical properties. As a result, Zr alloys are not acceptable for use as a fuel-cladding material in an SCWR.

As ferritic steels typically experience unacceptably high corrosion rates at the temperatures that will be present in an SCWR core [4,5], the prime candidate materials for in-core use in the various Generation IV SCWR concepts are austenitic stainless steels, with some consideration being given to nickel-based alloys. Austenitic stainless steels and nickel-based alloys were extensively evaluated in the various nuclear superheat programs carried out in the 1960s [6–8]; Ru and Staehle [9] have written an excellent overview of the US work. Indeed, many of the materials issues currently being studied in the various Generation IV SCWR research programs were identified and studied more than 50 years ago. There is also an extensive body of work on materials for use in FFSPs [10,11] and

* Corresponding author. Fax: +1 6135848219.
E-mail addresses: david.guzonas@cnl.ca (D. Guzonas), Sami.Penttila@vtt.fi (S. Penttilä), wcook@unb.ca (W. Cook), wenyue.zheng@canada.ca (W. Zheng), Radek.NOVOTNY@ec.europa.eu (R. Novotny), alberto.saez@ciemat.es (A. Sáez-Maderuelo), junya.kaneda.tv@hitachi.com (J. Kaneda).

http://dx.doi.org/10.1016/j.corsci.2016.01.034
0010-938X/

Please cite this article in press as: D. Guzonas, et al., The reproducibility of corrosion testing in supercritical water—Results of an international interlaboratory comparison exercise, *Corros. Sci.* (2016), http://dx.doi.org/10.1016/j.corsci.2016.01.034

supercritical water oxidation (SCWO), although the water chemistry in the latter application is much more corrosive than in a nuclear or fossil-fired power plant.

The Generation IV International Forum (GIF) SCWR Materials and Chemistry (M&C) Project Management Board (PMB) identified two major challenges that must be overcome to ensure the safe and reliable performance of an SCWR:

1. Insufficient data are available for any single alloy to unequivocally ensure its performance in an SCWR, especially for alloys to be used for in-core components.
2. Current understanding of SCW chemistry is inadequate to specify a chemistry control strategy, as the result of the large changes in physical and chemical properties of water through the critical point, coupled with the as yet poorly understood effects of water radiolysis.

To address these challenges, the GIF SCWR M&C Project Plan is made up of two work packages, one on SCWR Materials and the other on Radiolysis and Water Chemistry [12]. The Project Plan noted that:

> "*consideration should be given to sharing heats of materials to create a more consistent database. A selected number of heats of each alloy should be designated, and a plan developed for coordinating testing and characterization on these alloys by various participating organizations.*
>
> *It is proposed that round-robin testing and characterization of identical alloys under similar test conditions be carried out to assess the reproducibility of the results*. . ."

As a result, the GIF SCWR M&C PMB organized an interlaboratory comparison test (often called a round-robin test) to study the reproducibility of data obtained in different SCW test facilities on the general corrosion of un-irradiated candidate alloys in SCW.

The participants in the interlaboratory comparison test, and the designations used in this paper, were, by signatory:

Japan—Hitachi Research Laboratory (Hitachi);

Canada—Canadian Nuclear Laboratories (CNL),[1] CANMET Materials (CMAT), the University of New Brunswick (UNB);

EU Joint Research Centre—Institute for Energy and Transport (JRC-IET), VTT Technical Research Centre of Finland (VTT), Centro de Investigaciones Energéticas, Medioambientales y Tecnológicas (CIEMAT),[2] MTA Centre for Energy Research (MTA).[3]

As this was a collaborative effort within the Materials and Chemistry Project Arrangement, it was agreed by the participants that the tests would not start until the Project Arrangement was signed by all participants. The Materials and Chemistry Project Arrangement became effective in 2010 December, and the final planning of the tests was carried out during 2011, with actions to finalize the coupon dimensions, nominate the alloys and identify the amount required for the tests, and identify the participating facilities. The final test conditions were agreed to in 2012 January.

It was agreed that each participating signatory would provide coupons machined to the appropriate dimensions to the other participants, who would then follow a standard procedure to prepare (e.g., polishing) and characterize the coupons prior to testing, carry out the tests under the agreed upon test conditions, and then report the results. At the time of the signing of the Project Arrangement, the three signatories agreed to provide the following coupons (source organization in parentheses): Canada (CMAT)—Alloy 800H; EU (MTA)—08H18Ni10T stainless steel; Japan (Hitachi)—310 stainless steel.

The experiments were coordinated according to alloy, temperature regime, and major test parameters. It was decided to limit the data reporting to weight change only, due to differences in surface analysis and testing capabilities in different laboratories; participants were free to perform, and to report the results of, additional measurements if desired. In the context of this interlaboratory comparison test, weight change is defined as the final coupon weight after testing minus the initial coupon weight prior to testing. This is the number typically reported in general corrosion studies performed in support of the various SCWR concepts.

This paper presents the experimental procedure used for the tests, the results obtained, and discusses their significance.

2. Experimental

The compositions of the three alloys tested in the interlaboratory comparison tests are listed in Table 1.

Note that only a limited number of 310 SS coupons were available, and hence not all participants were able to test this material. The alloy 08H18Ni10T is equivalent to AISI 321 and DIN 1.4541.

2.1. Coupon preparation procedure

A coupon preparation procedure was developed and agreed to by the participants. The work flow and its division between coupon supplier and test participant are shown schematically in Fig. 1. The various steps in the work flow are described in detail below.

2.1.1. Alloy identification

The test alloys were to be identified by their commercial grade names. The name of supplier of test alloys as well as the chemical composition of the alloy was to be provided. All coupons were to be marked with a unique identifier. The coupon numbering scheme agreed to during the planning of the round-robin tests is summarized in Table 2.

2.1.2. Machining

The test coupons were to be machined into flat blocks with the dimensions 10 mm by 20 mm by 2 mm. A machining tolerance of 25 μm (1 mil) was considered acceptable. A hole measuring 5.5 mm in diameter was to be drilled at one end of the coupons for mounting. If necessary the hole size could be modified to facilitate mounting of the coupon in the test rig.

2.1.3. Sample size measurement

The dimensions of the samples were to be measured using a micrometer or a travelling microscope to an accuracy of 1 μm (10^{-3} mm) or better.

2.1.4. Polishing of the machined samples

Each test coupon was to be ground using 600 grit sand paper first, followed by 800 grit paper, and then with 1200 grit paper. A surface layer of at least 30 μm in thickness, as determined by measurement of the coupon dimensions before and after polishing, was to be removed from each side of the coupon to reduce the possible effects on corrosion of machining-induced plastic deformation at the metal surface.

2.1.5. Degreasing/cleaning

After surface finishing, the coupons were to be cleaned ultrasonically using acetone.

[1] Formerly Atomic Energy of Canada Limited.
[2] CIEMAT joined the test program after it had started and were only able to test one of the alloys.
[3] MTA later dropped out of the interlaboratory comparison tests.