Mechanical testing of anisotropy in ODS steel tubes

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Abstract. Anisotropy of fuel claddings made from Fe-9Cr and Fe-14Cr ODS (oxide dispersion-strengthened) steel for Generation IV reactors with coolants based on liquid metals was examined. Basic mechanical tests were performed at temperatures of 30 °C, 500 °C and 625 °C on ODS steel thin-walled tubes. Tensile tests were performed in axial and tangential directions in a vacuum to avoid oxidation of the small specimens. A scanning electron microscope with a small in-situ tensile testing device was used to conduct the tests. The result of this paper describes the basic mechanical properties of thin-walled tubes as ultimate tensile strength, yield strength, tensile modulus, etc. depending on the direction of loading.

1 Introduction

New types of Generation IV reactors are safer and more environmentally friendly than conventional reactors because they burn fuel more efficiently. These types include LFR (Lead-Cooled Fast Reactor System) reactors, which use heavy liquid metals (HLM) and their alloys (Pb, Pb-Bi, Pb-Li) as a coolant. Liquid metals contribute to safety through their internal properties, but they have a negative effect on construction materials because of the corrosive environment which they create.\footnote{[1]} One of the candidate materials for cladding fuel rods are ODS steels. ODS steels are strengthened by oxidic dispersions with elements Al, Ti, Y – there is a synergistic effect between the oxidic phase and the solid solution which improves the mechanical and physical characteristics of the steel. These steels have a longer life and much higher resistance to cyclic degradation, especially at high temperatures, than alloys without dispersed oxidic phase formation.\footnote{[1,2]}

ODS steels are products of powder metallurgy where the sintering powder itself has a significant impact on the resulting mechanical, corrosive and microstructural properties of the material. Due to the complicated manufacturing methods of ODS steels, it is important to check the required properties.

This paper deals with the investigation of the anisotropy of the mechanical properties of thin-walled tubes of Fe-9Cr and Fe-14Cr ODS steels which have been made for the fuel cladding for Generation IV reactors. Due to the limited amount of material in European laboratories and the small size of real components it is necessary to perform the material testing on miniature specimens. With regard to the anticipated operating temperatures, the tests must be done at temperatures up to 625 °C. Oxidation at high temperatures on miniature specimens of ODS steels changes their material characteristics, and therefore it is best to perform tests without air.\footnote{[3]}
2 Material

Two thin-walled tubes with diameters of approximately 10.7 mm and a wall thickness of approximately 0.6 mm were used for the experiments. The material of the first tube, with the designation K49 Fe-9Cr is powdered ferritic-martensitic steel with oxidic phases based on Y2O3. The second material, designated K48-T2-M3 Fe-14Cr is also ferritic-martensitic steel with a higher proportion of chromium containing Y2O3 oxides. Both materials show good resistance to creep at high temperatures. The chemical composition of the major alloying elements of the two materials are shown in Table 1.

| Designation (material) | Fe  [wt.%] | Cr  [wt.%] | W  [wt.%] | Ti  [wt.%] | Y  [wt.%] |
|------------------------|------------|------------|-----------|------------|----------|
| K49 (Fe-9 Cr)          | 88.1       | 9          | 2         | 0.51       | 0.3      |
| K48-T2-M3 (Fe-14 Cr)   | 84.4       | 14         | 1         | 0.26       | 0.3      |

3 Experiment

Miniature specimens for tensile testing in the axial and tangential direction of the ODS tube were made at Centrum Vyzkumu Rez (CVR) (Figure 1). The sample for tensile testing in the tangential direction is ring-shaped with a narrowing on the two opposite sides. The samples were dimensionally designed for Microtest 2000-EC, which is a part of the scanning electron microscope (SEM). The maximum allowable load of the testing device is 2 kN. The total initial cross-section area was designed to be approximately 1 mm² with regard to the maximum load capacity and to the expected ultimate strength of ODS steels. Considering the ODS tube thickness, 2 mm was selected as the width of the axial sample, and a width of 1 mm was chosen on each side in the tangential direction.

Tests were conducted in an SEM chamber at temperatures of 30 °C, 500 °C and 625 °C in a vacuum of 3·10⁻² Pa to avoid oxidation on the sample surface. The deformation speed of the crosshead was 0.4 mm/min (0.001 s⁻¹). Only the ultimate tensile strength (UTS) was measured on the samples in the tangential direction.

For samples in the axial direction, UTS and yield strength (YS), tensile modulus (E_mod), plastic elongation at break (A_utmn) and plastic elongation at UTS (Aₘ) were measured. To make this evaluation feasible, the sample was primarily designed with respect to the relationships between the initial cross-section and the initial measured length according to standard EN ISO 6892. The initial gauge length (L₀) was marked on the sample by using indentation imprints HV0.1. Measurement of the indentation distance was used instead of an extensometer. The measurement was made using SEM image analysis at selected load levels (see Figure 2).
4 Results

The resulting mechanical properties of the tensile test series performed on the ODS steel samples at temperatures of 30 °C, 500 °C and 625 °C are summarized in Table 2. $A_g$ and $A_{6mm}$ elongations are measured only in the axial direction. $A_{6mm}$ can be reliably evaluated only on samples where the break was in the centre of $L_0$. However, the samples often cracked beyond the middle third of $L_0$, therefore it was not possible to evaluate elongation at the break for all specimens and, particularly, there is no $A_{6mm}$ evaluation at high temperatures. $A_g$ is rounded to the nearest whole percent, and its character is purely informative, because its evaluation requires stopping the tension test at maximum load, but due to the flat load-displacement waveforms at UTS, such stopping is approximate, and so $A_g$-values are rounded to correspond to the accuracy of the measurement. Deformation measurement by SEM in the tangential direction on the ring shaped specimen was not possible, because the shape of the sample is complicated for both marking and mark observation. For the Fe-14Cr samples in the tangential direction at 500°C a more significant UTS decrease was observed than that of Fe-9Cr, and this corresponds to the appearance of the fracture surfaces (Figure 3).

![Figure 2. Comparison between elongations measured on $L_0$ (by SEM) and crosshead displacement.](image)

| Designation | Direction | Temperature (°C) | $E_{mod}$ (GPa) | YS (MPa) | UTS (MPa) | HV0.1 | $A_g$ (%) | $A_{6mm}$ (%) |
|-------------|-----------|-----------------|-----------------|----------|-----------|-------|----------|-------------|
| Fe-9Cr      | axial     | 30              | 226             | 829      | 956       | 364   | 8        | 14.8        |
|             | axial     | 500             | 118             | 499      | 637       | -     | 3        | -           |
|             | axial     | 625             | 78              | 316      | 455       | -     | 4        | -           |
|             | tangential| 30              | -               | -        | 933       | -     | -        | -           |
|             | tangential| 500             | -               | -        | 617       | -     | -        | -           |
|             | tangential| 625             | -               | -        | 424       | -     | -        | -           |
| Fe-14Cr     | axial     | 30              | 224             | 963      | 1049      | 373   | 7        | 16.8        |
|             | axial     | 500             | 152             | 652      | 737       | -     | 2        | -           |
|             | axial     | 625             | 99              | 442      | 558       | -     | 1        | -           |
|             | tangential| 30              | -               | -        | 996       | -     | -        | -           |
|             | tangential| 500             | -               | -        | 623       | -     | -        | -           |
|             | tangential| 625             | -               | -        | 494       | -     | -        | -           |
Conclusions
Mechanical tests on fuel cladding tubes made from Fe-9Cr and Fe-14Cr ODS steels show anisotropy between the axial and tangential directions (Table 2). The differences are higher for Fe-14Cr steel than that of Fe-9Cr steel. Moreover, a significant reduction of ductility occurs in Fe-14Cr steel at elevated temperatures. This reduction is most significant in the tangential direction at 500 °C.

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