Available online at www.sciencedirect.com
ScienceDirect
Journal of Nuclear Materials 356 (2006) 56–61
journal of nuclear materials
www.elsevier.com/locate/jnucmat

Tensile testing of EP-823 and HT-9 after irradiation in STIP II

Stuart A. Maloy [a,*], T. Romero [b], M.R. James [c], Y. Dai [d]

[a] *MST-8, MS H816, Los Alamos National Laboratory, Los Alamos, NM 87545, USA*
[b] *MS K575, NMT-11, Los Alamos National Laboratory, Los Alamos, NM 87545, USA*
[c] *MS G742, D-5, Los Alamos National Laboratory, Los Alamos, NM 87545, USA*
[d] *Paul Scherrer Institut, Spallation Neutron Source Division, CH-52, Villigen-PSI, Switzerland*

Abstract

The predicted operating conditions for a lead–bismuth eutectic target to be used in an accelerator driven system for the Advanced Fuel Cycle Initiative spans a temperature range of 300–600 °C while being irradiated by a high energy (~600 MeV) proton beam. Such spallation conditions lead to high displacement rates coupled with high accumulation rates of helium and hydrogen up to 150 appm/dpa. Two candidate ferritic/martensitic materials for these applications include HT-9 and EP-823. To investigate the effect of irradiation on these materials, the tensile properties were measured at 25, 250, and 400 °C after irradiation in the STIP II irradiation to doses up to 20 dpa at temperatures up to 350 °C. Losses in ductility concomitant with increases in yield stress are observed in both alloys although the embrittlement is more severe in the EP-823. This stronger embrittlement is attributed to the high silicon content in EP-823.
Published by Elsevier B.V.

---

1. Introduction

The Advanced Fuel Cycle Initiative is investigating different options for transmutation of high level nuclear waste. One of these options utilizes a high energy (~600 MeV) proton accelerator to bombard a lead bismuth eutectic target to produce a high energy neutron flux for transmuting fuel [1]. The structural materials used to contain this target will experience extensive radiation damage at operating temperatures of 400–600 °C. In addition, because the protons and neutrons will have energies greater than 20 MeV, spallation will occur causing a buildup of helium and possibly hydrogen (if the irradiation temperature is low enough) in the materials while displacement damage accumulates. To quantify the effect this environment has on the mechanical properties, materials are being irradiated at the Paul Scherrer Institut (PSI) under these prototypical conditions and the mechanical properties are being tested after irradiation.

Two ferritic/martensitic steels developed to withstand irradiation to high doses at temperatures from 400 to 600 °C are HT-9 and EP-823. HT-9 was developed in the breeder reactor program as a low swelling, high strength, ferritic/martensitic steel [2,3]. There have been many studies performed on the effects of irradiation on HT-9. This alloy was included to study the effects of high energy proton irradiation on its mechanical properties and as a

---

[*] Corresponding author. Tel.: +1 505 667 9784; fax: +1 505 667 7443.
E-mail address: maloy@lanl.gov (S.A. Maloy).

0022-3115/$ - see front matter Published by Elsevier B.V.
doi:10.1016/j.jnucmat.2006.05.003

reference alloy to compare against EP-823. EP-823 was developed in the Russian nuclear submarine program as a ferritic–martensitic steel with added silicon for improved corrosion resistance in lead bismuth. Thus, EP-823 is a candidate material for the accelerator-driven transmutation program.

To investigate the effects of high energy proton and neutron irradiation on the tensile properties of HT-9 and EP-823, the mechanical properties in tension were tested at 25, 250 and 400 °C. The experimental details are first described followed by the testing results and a discussion of these results. The major differences observed between HT-9 and EP-823 are described based on the differences in elemental content of the two ferritic/martensitic alloys.

2. Experimental details

EP-823 and HT-9 alloys were irradiated with following the chemical compositions:

- EP-823: 11.7Cr, 1.1Si, 0.73Mo, 0.63W, 0.65Ni, 0.54Mn, 0.34V, 0.26Nb and 0.17C.
- HT-9: 11.95Cr, 0.4Si, 1.0Mo, 0.6Ni, 0.6Mn, 0.3V, 0.2C.

The EP-823 was obtained from the Institute of Physics and Power Engineering (IPPE) in Obninsk, Russia. The materials were normalized at 1050 °C for 1 h and AC and tempered at 730 °C for 2 h and AC. The specimens had the dimensions of an S-1 tensile specimen which is a flat dogbone specimen 16 mm long, 4 mm wide, 0.75 mm thick with gauge dimensions of 1.2 mm × 5 mm. The alloys were irradiated in the STIP II irradiation under conditions listed in Table 1. The specimens were placed in Rods 1, 3, 7 and 10 in SINQ Target-4 (see Fig. 5 in Ref. [4]) to obtain different doses and temperatures. The irradiation temperature was monitored by a number of thermocouples installed in different rods in the target. The determination of temperature of each specimen has to rely on calculation. The STIP II target was modeled in MCNPX [5] to determine the fluences at various locations. Activation foils placed throughout the irradiation were used to verify the dose calculations using the computer code. The activities and relevant cross sections were input into the STAYSL2 [6] program along with computed estimates of the fluxes. The STAYSL2 program performed a least-squares regression fit to the data to create revised estimates of the proton and neutron fluxes. Only small changes to the neutron and proton fluxes were necessary to achieve good fits to the activation data. The irradiation data for the samples was computed based on a combination of the MCNPX calculations and results from the activation foils. For more information, see Ref. [4]. TEM specimens (placed near tensile specimens were) analyzed after irradiation and revealed helium contents ranging from 600 to 1500 appm He with a He/dpa ratio from 50 to 80 appm He/dpa. Hydrogen was observed at up to 700 appm for specimens irradiated below 155 °C, but no hydrogen was observed for specimens irradiated above 155 °C. Specimens were shipped from the Paul Scherrer Institut to Los Alamos National Laboratory using a Type 7A certified shipping container. Tensile testing was performed using an Instron test machine located in a hot cell. The test machine is equipped with a high temperature furnace capable of testing up to 700 °C in argon. Testing was performed at 20, 250 and 400 °C.

Table 1
STIP II irradiation conditions and tensile test conditions for EP-823 and HT-9 alloys

Material	Irr. temp (°C)	Dose (dpa)	Test temp (°C)
EP-823	140–340	7, 12, 18–19	20, 250, 400
HT-9	140–340	7, 12, 18–19	20, 250, 400

Testing was performed following ASTM E-8M-93. The initial strain rate was 5×10^{-4}/s. Specimens tested at elevated temperatures were held for 1 h to equilibrate before testing. The tensile test matrix is shown in Table 1. One or two specimens were tested in all conditions.

Engineering stress vs. engineering strain was calculated from the original gauge thickness, length and width (measured before testing) after subtracting the machine and fixture compliance from the load/displacement curves. These curves were used to determine 0.2% offset yield stress, ultimate tensile strength, uniform elongation and total elongation.

3. Results

Stress/strain curves for EP-823 and HT-9 tested at 25 °C are shown in Fig. 1(a) and (b). Significant differences are observed between the two alloys. EP-823 shows no plastic elongation after both irradiation doses at 13.3 and 16.2 dpa. These specimens broke in the elastic regime. Significant loss of ductility was also observed in HT-9 after irradiation but the specimens still retained some plastic ductility

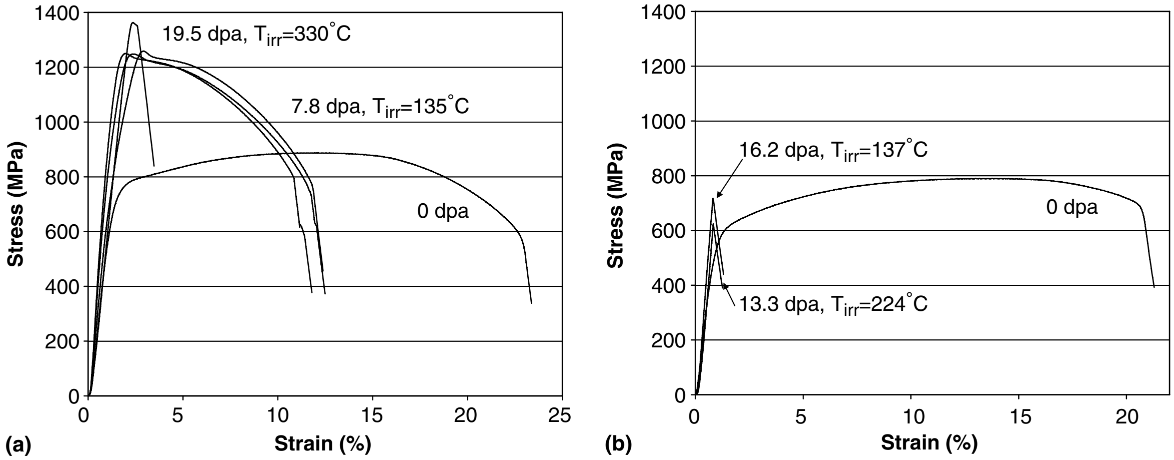

Fig. 1. Stress/strain curves measured on HT-9 (a) and EP-823 (b) at 25 °C after irradiation in STIP II.

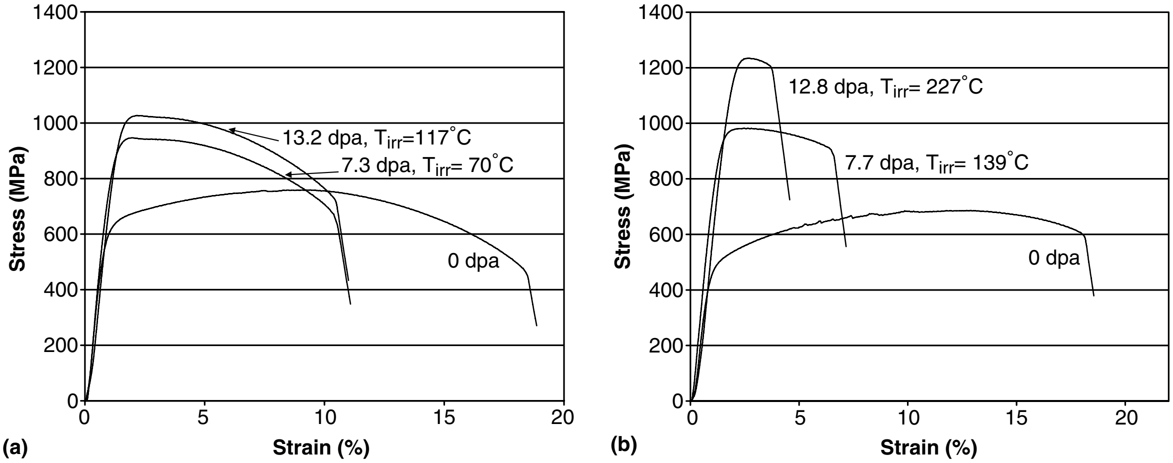

Fig. 2. Stress/strain curves measured on HT-9 (a) and EP-823 (b) at 250 °C after irradiation in STIP II.

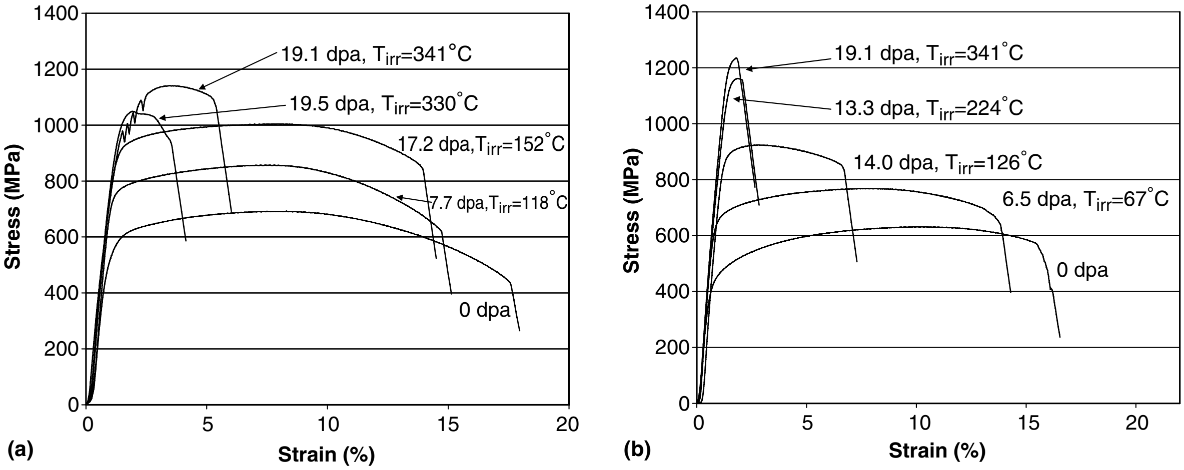

Fig. 3. Stress/strain curves measured on HT-9 (a) and EP-823 (b) at 400 °C after irradiation in STIP II.

for irradiations up to the highest dose, 19.5 dpa. Because yielding was observed in the HT-9 specimens, one could also observe the increase in yield stress with dose which could not be measured for the EP-823 specimens.

Stress/strain curves for EP-823 and HT-9 for testing at 250 °C are shown in Fig. 2(a) and (b). 250 °C was chosen as a test temperature because it was one of the main reference test temperatures for all testing from the STIP irradiations. Embrittlement is not as severe as observed for the testing at 20 °C but similar trends are observed when comparing the properties measured on EP-823 to those measured on HT-9. A larger increase in yield stress is observed for EP-823 and a more significant change in uniform and total elongation.

Fig. 3 shows stress/strain curves for HT-9 (a) and EP-823 (b) tested at 400 °C. These specimens were irradiated to the highest dose, 19.5 dpa, of all specimens tested. At the highest dose, both EP-823 and HT-9 show large reductions in elongation and significant increases in yield stress. Once again, a larger reduction in ductility and increase in yield stress is observed for EP-823. In addition, load drops were observed in the plastic portion of the stress/strain curve for HT-9 irradiated to 19.5 dpa.

4. Discussion

A compilation of the change in yield stress and uniform elongation with dose for both HT-9 and EP-823 are shown in Fig. 4. This data shows the lack of uniform elongation observed for EP-823 when tested at room temperature after irradiation and a strong reduction in uniform elongation with dose for both HT-9 and EP-823. The plot showing the change in yield stress with dose (Fig. 4(b)) elucidates the decrease in yield stress with increasing temperature for both alloys and a stronger increase in yield stress with increasing dose for EP-823 over HT-9 especially for doses above 10 dpa. One exception to this trend is observed for the two points labeled as fracture stresses which were measured at room temperature for specimens breaking in the elastic regime.

The effects of irradiation temperature on the tensile properties of HT-9 and EP-823 are somewhat hidden in the previously discussed data. The irradiation temperature ranged from 67 to 340 °C. In general, the highest dose specimens were at the highest irradiation temperatures. Although, some rods (holding specimens) were irradiated at lower overall temperatures than other rods. The most significant effect of irradiation temperature is observed in the specimens tested at 400 °C (Fig. 3). In this case, the test temperature was higher than the irradiation temperature. The stress/strain curves for the specimens with the lowest irradiation temperature showed good ductility in the stress/strain curves while the specimens with a higher irradiation temperature (above 200 °C) showed reduced ductility after irradiation. This suggests that testing at

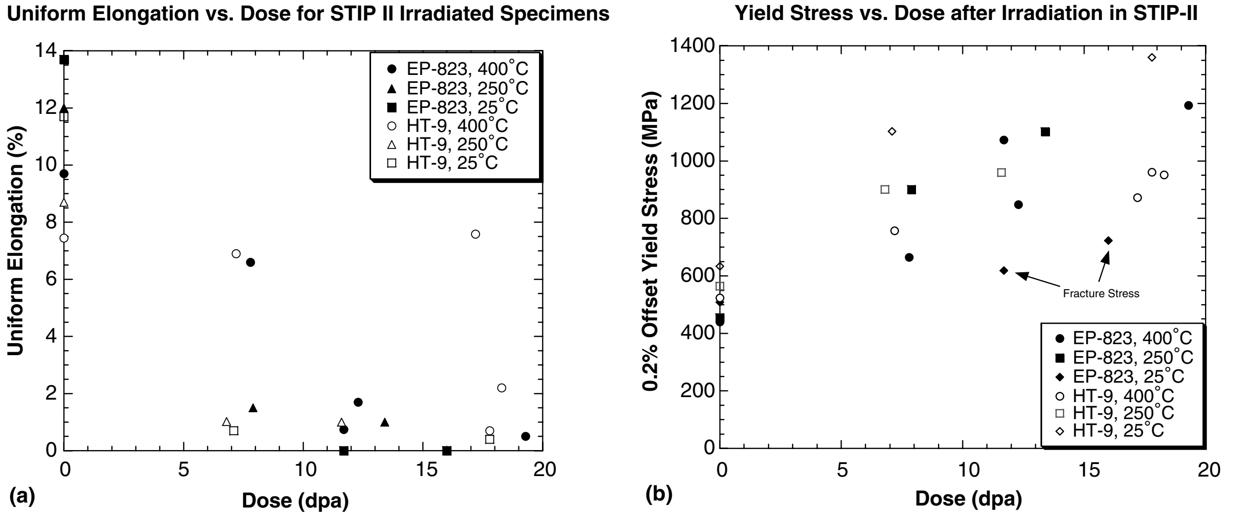

Fig. 4. Graphs showing the changes in uniform elongation (a) and 0.2% offset yield stress (b) vs. dose for HT-9 and EP-823 after exposure in the STIP-II irradiation.

400 °C led to some annealing or rearrangement of the of the defect microstructure produced at <150 °C resulting in a higher uniform elongation in the stress/strain curves. Interestingly, an increase in yield stress was still observed in the stress/strain curves. These results are similar to those of Chen et al. (this conference and [7]) where annealing was performed after irradiation at various temperatures up to 700 °C. EP-823 exhibits a lower elongation than HT-9 when the specimens irradiated at lower temperatures are tested at 400 °C.

The previously described results for EP-823 point out a stronger embrittling response to irradiation when compared to HT-9. EP-823 shows fracture in the elastic regime at room temperature, a continued increase in yield stress with increasing dose and less of an improvement in ductility when tested at 400 °C. The most significant difference between EP-823 and HT-9 is its increased silicon content and the small tungsten and niobium additions. Previous work on silicon containing ferritic/martensitic steels showed aging at temperatures from 500 to 600 °C led to a decrease in toughness with increasing silicon content caused by precipitation of Laves-phases containing Si and P [8,9]. Such precipitation has also been observed under irradiation at 400 °C [10,11]. Similar embrittlement was not observed in reduced activation alloys containing tungsten with very low silicon contents [12]. In addition, fission reactor irradiations on EP-823 led to embrittlement for irradiations at temperatures below 460 °C [13].

Because of the high energy spectrum in the STIP II environment, large amounts of spallation-produced gases buildup in these specimens during irradiation. The high irradiation temperatures allow most of the hydrogen to escape while not affecting the helium. Helium accumulates at a rate of 50–80 appm/dpa. Thus, the highest dose specimens have up to 1400 appm helium. Although there is a large database on the effects of fission reactor irradiation on the mechanical properties of HT-9, the properties measured are strongly affected by irradiation temperature and no data exists for irradiations at 340 °C to compare to irradiation data for this irradiation. The most similar irradiation conditions found were those from an irradiation in FFTF at 360 °C and tested at 205 °C. In this case the yield measured was 1005 MPa after irradiation to a dose of 40 dpa [14]. This compares well with our yield of 1025 MPa measured at 250 °C after irradiation to 13.2 dpa at an irradiation temperature of 117 °C assuming that the increase in yield stress with dose increases very slightly at doses above 10 dpa.

5. Conclusions

Tensile testing has been performed on EP-823 and HT-9 after irradiation in the STIP II irradiation at the Paul Scherrer Institut. Specimens were irradiated to a total dose of up to 19.3 dpa at temperatures up to 340 °C and a significant effect of irradiation is observed on the tensile properties. The yield stress increases with dose out to 20 dpa. Testing at increasing temperature from 25 to 400 °C results in slight decreases in yield stress and increasing uniform elongation. Stronger embrittlement is observed in EP-823 compared to HT-9. EP-823 specimens broke in the elastic regime at room temperature after irradiation. The yield stress increases more steeply with dose in EP-823. These results suggest a strong effect of silicon on mechanical properties of EP-823.

Acknowledgements

The authors would like to acknowledge Brian Oliver of Pacific Northwest National Laboratory for performing the hydrogen and helium measurements after irradiation. Thanks are also due to A. Russanov of IPPE for providing the EP-823 material for this irradiation. This work was performed under the auspices of the Advanced Fuel Cycle Initiative program for the Department of Energy.

References

[1] S. Wender, Preliminary assessment of spallation target options for accelerator-driven transmutation. AAA-RPO-TRNS-01-0017, LAUR-01-1634, Los Alamos National Laboratory, Los Alamos, NM, 2001, p. 20.
[2] W. Bell, T. Lauritzen, S. Vaidyanathan, in: Proceedings for Topical Conference on Ferritic Alloys for Use in Nuclear Energy Technologies, Snowbird, Utah, Metallurgical Society of AIME, 1984, ISBN 0-89520-458-4, p. 113.
[3] T. Lauritzen, W. Bell, S. Vaidyanathan, in: Proceedings for Topical Conference on Ferritic Alloys for Use in Nuclear Energy Technologies, Snowbird, Utah, Metallurgical Society of AIME, 1984, ISBN 0-89520-458-4, p. 623.
[4] Y. Dai, X. Jia, R. Thermer, D. Hamaguchi, K. Geissmann, E. Lehmann, H. Linder, M. James, F. Groschel, W. Wagner, G. Bauer, J. Nucl. Mater. 343 (1–3) (2005) 33.
[5] MCNPX Version 2.4.0 User's Manual, LA-CP-02-408, August 2002.
[6] M.R. James, S.A. Maloy, J.W.F. Sommer, P.D. Ferguson, M.M. Fowler, G.E. Mueller, R.K. Corzine, in: J.G. Williams

et al. (Eds.), Reactor Dosimetry: Radiation Metrology and Assessment, ASTM, West Conshohoken, PA, p. 167.

[7] J. Chen, M. Rodig, F. Carsughi, Y. Dai, G. Bauer, H. Ullmaier, in: Proceedings of the 6th International Workshop on Spallation Materials Technology IWSMT-6, August 1, 2005, vol. 343, p. 236–240, J. Nucl. Mater. 343 (1–3) (2005) 236.

[8] Y. Hosoi, N. Wade, S. Kunimitsu, T. Urita, J. Nucl. Mater. 141–143 (PTA) (1986) 461.

[9] Y. Hosoi, N. Wade, T. Urita, M. Tanino, H. Komatsu, J. Nucl. Mater. 133&134 (1985) 337.

[10] H. Kawanishi, R. Hajima, N. Sekimura, Y. Arai, S. Ishino, J. Nucl. Mater. 155–157 (1988) 887.

[11] T. Yukitoshi, K. Yoshikawa, H. Teranishi, T. Lauritzen, W. Bell, S. Vaidyanathan, J. Nucl. Mater., in press.

[12] S. Maloy, M. James, T. Romero, M. Toloczko, R. Kurtz, A. Kimura, J. Nucl. Mater. 341 (2–3) (2005) 141.

[13] S. Porollo, A. Dvoriashin, Y. Konobeev, F. Garner, J. Nucl. Mater. 329–333 (2004) 314.

[14] F.H. Huang, Fracture toughness and tensile properties of alloy HT9 in thin sections under high neutron fluences, 1125, ASTM Special Technical Publication, 1992, 1267.