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HEDL--7421

DE84 005381

SWELLING IN SEVERAL COMMERCIAL
ALLOYS IRRADIATED TO VERY HIGH
NEUTRON FLUENCE - CONTRIBUTION
TO DOE/ER-0045-11

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HANFORD ENGINEERING DEVELOPMENT LABORATORY
Operated by Westinghouse Hanford Company, a subsidiary of
Westinghouse Electric Corporation, under the Department of
Energy Contract No. DE-AC06-76FF02170
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7.1 SWELLING IN SEVERAL COMMERCIAL ALLOYS IRRADIATED TO VERY HIGH NEUTRON FLUENCE - D. S. Gelles and J. S. Pintler (Westinghouse Hanford Company)

7.1.1 ADIP Task

The Department of Energy (DOE)/Office of Fusion Energy (OFE) has cited the need to investigate ferritic alloys under the ADIP program task, Ferritic Steels Development (Path E). The tasks involved are akin to task number 1.C.2, Microstructures and Swelling in Austenitic Alloys and task number 1.C.1, Microstructural Stability.

7.1.2 Objective

The objective of this work is to provide guidance on the applicability of martensitic stainless steels for fusion reactor structural components.

7.1.3 Summary

Swelling values have been obtained from a set of commercial alloys irradiated in EBR-II to a peak fluence of 2.5×10^{23} n/cm² ($E > 0.1$ MeV) or ~ 125 dpa covering the range 400 to 650°C. The alloys can be ranked for swelling resistance from highest to lowest as follows: the martensitic and ferritic alloys, the niobium based alloys, the precipitation strengthened iron and nickel based alloys, the molybdenum alloys and the austenitic alloys.

7.1.4 Progress and Status

7.1.4.1 Introduction

Irradiation induced swelling is an important materials property affecting the design of a fusion reactor. Swelling must be compensated for when determining design tolerances and is often found to be a limiting design factor. Unfortunately, generation of swelling information for very high fluence neutron irradiation requires long irradiation times and it is rarely possible to perform such experiments. As part of the National Cladding/Duct Materials Development Program (NCD) for Liquid Metal Fast Breeder Reactor development, a series of commercial alloys was included in the AA-I swelling test and irradiations were begun in December 1974 in the Experimental Breeder Reactor (EBR) II, at Idaho Falls, ID. As these materials are no longer being studied by the NCD program, they have been made available for fusion related studies. Results of density change measurements on specimens examined at earlier discharges of the AA-I test have been reported previously.^{1,2} The purpose of the present effort is to provide results on specimens irradiated in the final irradiation of the AA-I test to a peak fluence of 2.53×10^{23} n/cm² (all fluences are reported as $E > 0.1$ MeV) or approximately 125 dpa.

The specimens which were included in the AAI test covered a large number of different types of commercial alloys. A listing of alloy compositions is provided in Table 7.1.1. Several alloy classes are represented. Six ferritic and martensitic alloys covered the composition range Fe-5Cr to Fe-22Cr and included carbide, yttria and copper precipitate strengthening. Three iron base solid solution strengthened alloys, one with high silicon additions, provided results for the Fe-(20 to 35) Ni - (20 to 25) Cr composition range. Nine precipitation strengthened alloys in the superalloy class were included covering both the iron-and nickel-base range. Both Ni₃(Al,Ti) and Ni₃Nb precipitate strengtheners were included. Four nickel base solid solution strengthened alloys, most with high molybdenum contents, were irradiated as were two refractory alloys, one a niobium base alloy and the other a molybdenum base alloy. Therefore, the AAI test was able to provide results of irradiation induced swelling for a wide range of commercial structural alloys.

7.1.4.2 Experimental Procedure

Details of the AAI test design and configuration have been documented previously.^{1,2} The present effort concerns capsule B116 following its removal from reactor for the fourth time. Examinations of AAI specimens generally involved measurements on specimens which had not been measured at a previous discharge. (One exception occurred in the case of specimens irradiated at 590°C to a fluence of 1.78×10^{23} n/cm².) This procedure was possible because the original AAI test involved side-by-side irradiation of four identical capsules, and only for one case was it necessary to measure specimens which had been measured previously and then reirradiated. Earlier removals were required in order to replace the stainless steel hardware (capsule and subcapsules) which were beginning to swell and thereby altering the operating temperatures. It was also found that the actual operating temperatures were lower than anticipated due to lower than expected gamma heating in EBR-II throughout the life of the test. The temperatures reported in this paper as in references (1) and (2) are the design temperatures. The actual temperatures are as much as 20°C lower for the highest temperature subcapsules and proportionately less for the remainder.

The compositions of the specimens examined from the AAI test are listed in Table 7.1.2 and the heat treatment given these specimens are provided in Table 7.1.2. Composition overchecks by Lukens Steel Company are in good agreement with the values in Table 7.1.1. It was apparent that a great deal of effort would be required to measure the density change of every specimen in capsule B116 of the AAI test and therefore only a limited number of specimens was selected for density change measurements. Those specimens of major interest to the Fusion materials community, namely those ferritic alloys and refractory alloys

which were previously examined,² were measured for density following irradiation at each irradiation temperature where available. The remainder was only measured after irradiation at two temperatures, 425°C and 540°C. These two were selected because they corresponded to high flux conditions. The 425°C condition was intended to identify materials which develop a swelling maximum at low temperatures. The 540°C condition was intended to provide data at a moderately high temperature, close to the peak swelling temperature for AISI 316 stainless steel.³ Details regarding density measurement procedures have been described previously.^{1,2}

Table 7.1.2 Swelling measurements for specimens irradiated in B116 of the AA-I test

Alloy	Heat Treatment	Temperature (°C) Fluence (10 ²³ n/cm ²)	Swelling (ΔV/V ₀ , %)							
			400 1.60	425 2.07	450 1.55	480 1.98	510 2.41	540 2.32	590 2.53	650 2.50
H11	1010/1/W.Q.+570/2/W.Q.		0.16	0.14	0.18	-0.11	-0.05	0.04		
EM12	1050/0.5/A.C.+750/1.5/A.C.		0.57	0.56	-0.02	-0.28	-0.38	-0.29		0.08
AISI 416	870/F.C. AT 13°C Per Hour to 590/A.C.		0.35	0.25	0.08	0.08	0.20	0.05		0.21
430 F	1070/1/W.Q.		0.23	0.20	0.09	0.17	0.24	0.05		0.11
FeCrAlY	Consolidated at 1150°C			0.12				-0.18		
C.C. 455	1070/1/W.Q. + 510/4/A.C.			-0.36				1.78		
AISI 310	1070/1/W.Q.			49.79				17.92		
RA-330	1070/1/W.Q.			12.36				2.04		
INC. 800	1070/1/W.Q.			41.08				11.95		
A-286	1070/1/W.Q.			31.60				0.51		
A-286	980/1/O.Q. + 720/16/A.C.			N.M.				0.81		
M813	1080/4/A.C. + 900/1/A.C. + 750/8/A.C.			1.75				1.84		
D979	1020/2/W.Q. + 840/6/A.C. + 705/16/A.C.			-1.91				-2.45		
INC. 901	1100/3/W.Q. + 790/4/A.C. + 720/24/A.C.			0.15				-0.07		
INC. 718	750/1/W.Q. + 720/8/F.C. to 620/18 total A.C.			0.60				0.25		
INC. X-750	1150/2/A.C.			1.06				-0.06		
INC. X-750	1150/2/A.C. + 840/24/A.C. + 700/20/A.C.			4.18				0.46		
INC. X-750	1150/2/A.C. + 840/0.5/A.C.			1.47				-0.05		
NIM. 80A	1080/8/A.C. + 705/16/A.C.			1.16				0.33		
NIM115	1190/1.5/A.C. + 1100/6/A.C.			2.05				7.17		
INC 625	1150/1/W.Q.			1.01				-0.06		
HAST X	1190/1/W.Q.			-2.30				38.00	79.90	
HAST S	1070/1/A.C.			-0.30				0.06		
HAST C-4	1070/1.5/W.Q.			-0.09				4.79		
INC 600	1120/1.5/A.C.			12.70				1.60		
INC 600	20% cold worked			7.92				0.07		
Nb-1Zr	1200/1/V.C.		0.43	0.53	0.25	0.16	0.43	0.31	0.39	0.56
TZM	1300/2.5/V.C.		0.35	0.03	0.03	0.02	0.36	0.05	3.77	2.86

Heat Treatment Code: temperature (°C)/time (hour)/W. Q. = water quench, A.C. = air cooled, F.C. = furnace cool, O.Q. = oil quench, V.C. = cool under vacuum.

7.1.4.3 Results

The swelling results are presented in Table 7.1.2. Results in Table 7.1.2 for several of the ferritic alloys and for the refractory alloys which were measured over the full range of irradiation temperature show that these materials are low swelling alloys. Only TZM developed swelling in excess of one percent. Results shown for the remaining alloys indicate that most of the alloys investigated are low swelling alloys, but several notable exceptions occur. In interpreting these results, it should be noted that swelling and density change are not equal at high values. Volumetric swelling in percent, ΔV/V₀, must be computed according to the relationship:

$$S(\%) = \frac{(\Delta\rho/\rho_0)}{1 - (\Delta\rho/\rho_0)} \times 100$$

where $\frac{\Delta\rho}{\rho_0}$ is the fractional density change.

For example, the density change value of 33.24 percent for AISI 310 irradiated at 425°C corresponds to 49.8 percent swelling. The remainder of this section will be a description of each alloy and its swelling response.

Inconel 625, a nickel base gamma double prime strengthened alloy. The swelling response of Inconel 625 was similar to that of X-750 and Nimonic 80A. The swelling was 1.00 percent at 425°C and -0.06 percent at 540°C. In summary, iron and nickel base precipitation strengthened superalloys are generally low swelling materials. The exceptions either involved a situation where a phase change promoted high swelling or where an overaged microstructure was used and moderate swelling developed.

Nickel Base Solid Solution Strengthened Alloys

Hastelloy X is a high-temperature corrosion resistant alloy with approximately 20 percent chromium and 20 percent iron. Hastelloy X was found to densify, -2.36 percent, following irradiation at 425°C and swell 38.0 percent at 540°C. Density change measurements on Hastelloy X showed 44.4 percent density change at 590°C following irradiation to 2.47×10^{23} n/cm². This corresponds to 79.9 percent swelling. Therefore, solid solution hardened alloys in the 50 percent nickel range can develop very high swelling and the peak swelling temperature is high. In comparison, Hastelloy S, a similar alloy with negligible iron and higher levels of molybdenum and nickel, was highly swelling resistant. The swelling was -0.30 percent at 425°C and 0.06 percent at 540°C. Hastelloy C-4 a similar alloy gave intermediate results. At 425°C, -0.09 percent swelling was found but at 540°C, 4.79 percent swelling developed. Inconel 600, an alloy intermediate in composition between Hastelloy X and Hastelloy S or C-4 but without molybdenum and with minor additions of gamma prime forming elements, developed moderate swelling. A value of 12.7 percent swelling was found at 425°C and 1.60 percent for 540°C in the case of solution annealed Inconel 600. Inconel 600 in the 20% cold worked condition developed 7.92 percent swelling at 425°C and 0.07 percent swelling at 540°C. In summary, nickel based solid solution hardened alloys developed a wide range of swelling responses. Low swelling, moderate swelling and high swelling alloys were found and the temperature dependence of swelling varied from peak swelling at low temperatures to peak swelling at high temperatures. An explanation for this wide variation in response is not yet available.

Refractory Alloys

Nb-1Zr is a commercial niobium base alloy used in the body centered cubic state for high temperature applications. It is found to be highly swelling resistant, at least over the temperature range studied in the experiment, 400 to 630°C. Comparison with lower fluence data² appears to show that a larger scatter in the data occurs at higher fluence and that swelling in this alloy has saturated. TZM is a commercial molybdenum base body centered cubic alloy used for high temperature applications. It is found to develop moderate swelling over the available range of irradiation temperature with peak swelling of 3.77 percent measured for the 590°C irradiation condition.

This represents a low swelling rate, 0.07 percent increase over 7.5×10^{22} n/cm² (Ref. 2) or .002 percent per dpa, and indicates that the swelling of this alloy is approaching saturation. A similar comparison for the 630°C case shows that TZM is densifying at high fluence. A specimen to specimen variation might explain the difference but certainly initial predictions based on lower fluence response⁶ cannot be justified. In summary, refractory commercial alloys are moderately to highly swelling resistant with peak swelling occurring at 590°C. Saturation is apparent in both alloys examined at fluence levels on the order of 2.0×10^{23} n/cm² or 100 dpa.

Discussion

Based on this study of density change in a series of commercial alloys irradiated to fluences as high as 2.5×10^{23} n/cm² or 125 dpa, it is possible to draw several generalizations regarding swelling in commercial structural alloys at high fluence in a fast neutron environment. This section is intended to provide recommendations regarding the more swelling resistant alloy classes, a basis for understanding the response by comparison with the response of simple experimental alloys, and a commentary on effects which arise due to overaging in precipitation strengthened alloys and minor element additions.

The Low Swelling Alloy Classes

The present work identifies three classes of commercial alloys which remain either low swelling (less than 1 percent) or moderate swelling (less than 5 percent) at high fluence. Ferritic/Martensitic alloys are found to be the most swelling resistant as a group of alloys. Apart from the case where austenite formation is expected to have influenced behavior, swelling remains well below one percent and the highest swelling alloy maintains a very low swelling rate: 0.05 percent per 10^{22} n/cm² or 0.01 percent per dpa. In comparison, refractory alloys are less swelling resistant but still low swelling. However, the refractory alloy class of commercial alloys does develop significant differences in swelling level from one alloy to another: 0.6 percent for Nb-1Zr versus 3.6 percent for TZM. The alloy class does appear to develop swelling saturation at doses on the order of 100 dpa and therefore further increases in swelling are not expected at even higher doses. Precipitation strengthened iron and nickel base superalloys are also found to be low or moderate swelling except in the exceptional cases where a phase transformation or overaging is involved. However, saturation cannot be generally demonstrated in this alloy class and therefore higher swelling can be anticipated at still higher doses.

The results tend to follow predictions based on the response of simple alloys but many exceptions are found which demonstrate that minor element additions and microstructure can play an important role in the control of swelling.

7.1.6 Further Work

Microstructural examinations on selected specimens will be performed in the next reporting period.

7.1.7 References

1. J. F. Bates and R. W. Powell, "Irradiation-Induced Swelling in Commercial Alloys," *J. Nuc. Mat.* 102 (1981) 200.
2. R. W. Powell, D. T. Peterson, M. I. Zimmerscheid and J. F. Bates, "Swelling in Several Commercial Alloys Following High Fluence Neutron Irradiation," *Ibid*, 103 & 104 (1981) 969.
3. J. F. Bates and M. K. Korenko, "Empirical Development of Irradiation-Induced Swelling Design Equations," *Nuc. Tech.* 48 (1980) 303.
4. D. S. Gelles, "Microstructural Examination of Several Commercial Ferritic Alloys Irradiated to High Fluence," *J. Nuc. Mat.* 103 & 104, (1981) 975.
5. D. S. Gelles, L. E. Thomas and J. J. Laidler, "Swelling in Previously Neutron Irradiated Commercial Fe-Cr-Ni Based Alloys Under Electron Irradiation," *J. Nuc. Mat.* 108 & 109 (1982) 504.
6. D. S. Gelles, D. T. Peterson and J. F. Bates, "Void Swelling in the Molybdenum Alloy T2M Irradiated to High Fluence," *J. Nuc. Mat.* 103 & 104 (1981) 1141.
7. D. S. Gelles and L. E. Thomas, "Effects of Neutron Irradiation on Microstructure in Experimental and Commercial Ferritic Alloys," HEDL-SA-2772FP, to be published in the *Proceedings of the AIME Topical Conference on Ferritic Alloys for Use in Nuclear Energy Technologies*.
8. F. A. Garner, "Dependence of Swelling on Nickel and Chromium Content in Fe-Ni-Cr Ternary Alloys," *Damage Analysis and Fundamental Studies Quarterly Progress Report*, DOE/ER-0046/14 (July 1983) 133.
9. H. R. Brager, "Swelling of High Nickel Fe-Ni-Cr Alloys in EBR-II," *Ibid*.
10. J. F. Bates, "Irradiation-Induced Swelling Variations Resulting from Compositional Modifications of Type 316 Stainless Steel," *Properties of Reactor Structural Alloys after Neutron or Particle Irradiation*, ASTM STP 570 (1975) 369.
11. J. F. Bates, R. W. Powell and E. R. Gilbert, "Reduction of Irradiation-Induced Creep and Swelling in AISI 316 by Compositional Modifications," in *Effects of Radiation on Materials: Tenth Conference*, ASTM STP 725 (1981) 713.
12. H. R. Brager and F. A. Garner, "Radiation-Induced Evolution of the Austenitic Matrix in Silicon - Modified AISI 316 Alloys," in *Phase Stability During Irradiation*, AIME (1981) 219.
13. F. A. Garner, "The Microchemical Evolution of Irradiated Stainless Steels," *Ibid*, 165.