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Journal of Nuclear Materials 271&272 (1999) 370–375

journal of nuclear materials

Effect of temperature change on void swelling in P, Ti-modified 316 stainless steel

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Abstract

Irradiation experiment on 20% cold worked P, Ti-modified 316 stainless steel with step-wise change of temperature was conducted in JOYO and Phenix. These were analyzed from the viewpoint of the influence of temperature change on microstructure change. With temperature decreasing, void formation is accelerated, compared with that of with constant temperature irradiation, and void formation is suppressed as temperature increases. From these results, it is suggested that the change of void formation behavior is caused by step-wise change of vacancy supersaturation associated with a change in irradiation temperature. © 1999 Elsevier Science B.V. All rights reserved.

1. Introduction

Type 316 stainless steel was an early choice for the candidate materials not only in the fast reactor development program but also in the fusion program. A 20% cold worked P, Ti-modified 316 stainless steel was commercially developed for utilization as in-core materials in the fast reactor program of PNC, in order to attain the dimensional stability of subassemblies at high neutron fluence [1].

It is experimentally known that the void swelling is significantly affected by the temperature change under neutron irradiation, based on the microstructural observation of neutron irradiated samples (e.g. [2]). Those studies are very important in the light of the first wall of fusion reactor with cyclical temperature change and also for fast reactor fuel assembly subject to temperature changes due to burning of MOX fuel and by shuffling of the assembly.

In this paper the effects of irradiation temperature change on microstructural evolution of 20% cold worked P, Ti-modified 316 stainless steel irradiated with a step-wise change of temperature are discussed.

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2. Experimental procedure

Fuel cladding used in this experiment were made for two lots of P, Ti-modified 316 stainless steel. The chemical composition, solution annealing condition and cold work level on each steel are listed in Table 1. Outer diameter and wall thickness of cladding before irradiation were 6.5 and 0.47 mm, respectively.

The L1 lot of cladding was irradiated in JOYO using Core Material Irradiation rig (CMIR) with a step-wise decrease of irradiation temperature. The details of temperature change are shown Table 2. The specimens were at first irradiated at constant temperature during CMIR-1–CMIR-3. After the end of CMIR-3 irradiation, the specimens irradiated at 863 and 753 K were cut in half. During the CMIR-4 irradiation, one half of each specimen continued to be irradiated at the original temperatures and the other half of each specimen was irradiated at lower temperature of 713 K.

The coupling irradiation in Phenix/JOYO was carried out using the different lot L2 of cladding using Phenix's sample irradiation rig and CMIR, details of which are also shown in Table 2. During CMIR irradiation, the irradiation temperature of each cladding was increased by 20–50 K above the temperature of irradiation in Phenix.

The swelling of the irradiated cladding was measured between intervals of each irradiation period by means of either diameter determination or density measurement. TEM observation was also carried out for some speci-

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Table 1
Chemical composition and fabrication condition of cladding lots

Cladding lot No.	Chemical composition (wt%)											Final solution annealing condition	Cold work level (%)
	C	Si	Mn	P	Ni	Cr	Mo	B	Ti	Nb + Ta	Fe		
L1	0.062	0.82	1.95	0.028	13.77	16.70	2.34	0.005	0.084	0.042	bal.	1353 K × 1 min	20
L2	0.047	0.92	1.74	0.030	13.74	16.50	2.50	0.004	0.097	0.070	bal.	1358 K × 1 min	19

Table 2
Irradiation condition, types of post-irradiation examination and value of swelling

	Irradiation period	CMIR-1	CMIR-2	CMIR-3	CMIR-4
Temperature change irradiation	713K	2.6 Diameter -0.09	6.7 Diameter -0.18	9.8 Diameter,TEM -0.18	12.7 Diameter,TEM -0.16
	753K	2.5 Diameter -0.09	6.4 Diameter -0.18	9.4 Diameter,TEM -0.15	12.2 Density,TEM -0.36
	753K/713K			→	12.3 Density,TEM -0.25
	863K	3.4 Diameter -0.09	8.8 Diameter -0.15	12.9 Diameter,TEM -0.09	16.7 Density,TEM -0.03
	863K/713K			→	15.8 Density,TEM 2.77
Coupling irradiation	Irradiation period	Phenix	CMIR-2	CMIR-3	CMIR-4
	753K/773K	16.6 Density,TEM 0.00	22.0 Density,TEM 0.40	26.1 Density 1.80	29.9 Density,TEM 3.93
	793K/843K	18.9 Density,TEM -0.04	25.4 Density,TEM 0.10	30.3 Density,TEM 0.60	34.9 Density,TEM 2.17
	843K/873K	18.5 Density,TEM 0.06	24.9 Density,TEM 0.20	29.7 Density,TEM 0.60	34.2 Density,TEM 1.20

The Upper row: Fluence ($\times 10^{26}$ n/m^2; $E > 0.1$ MeV)
The Middle row: PIE items
The Lower row: Value of swelling (%)

mens which were cut off from irradiated cladding at the end of each irradiation period.

3. Results

3.1. Swelling measurement

The results of swelling measurements of the irradiated claddings are shown in Table 2. In the step-wise temperature change irradiation program using CMIR, no swelling occurred for claddings irradiated at constant temperature. The specimens changed from 753 to 713 K during the CMIR-4 period showed on abrupt jump in swelling as compared with the constant temperature specimens. For the specimens changed from 753 to 713 K, the swelling increase was negligible as compared with specimens irradiated at constant temperature of 753 or 713 K.

On the other hand, in the coupling irradiation program in Phenix and JOYO, influence of reactor and temperature change on swelling was not clear.

3.2. TEM observation

Fig. 1 shows microstructures of P, Ti-modified 316 stainless steels irradiated in the step-wise temperature change irradiation program using CMIR. At the end of CMIR-3 irradiation period, void formation had not occurred in any specimen. Formation of needle-shaped phosphides had occurred in some specimens irradiated at 863 and 753 K. Formation of other precipitates was not observed in any specimens. At the end of the CMIR-4 irradiation period, voids were not observed in the constant temperature specimens at 863 and 713 K, and a few voids formed in the constant temperature specimen at 753 K. Some precipitates were formed in the 863 and 753 K specimens. These are as identified M_6C, Laves and phosphides in the 863K specimen, and M_6C and phosphide in the 753 K specimen. In the 713 K specimen, formation of M_6C precipitates had occurred to a small extent. In the step-wise temperature change specimens, a lot of voids are formed. Void formation in temperature changed specimen from 863 to 713 K is more pronounced than in the specimen dropping from

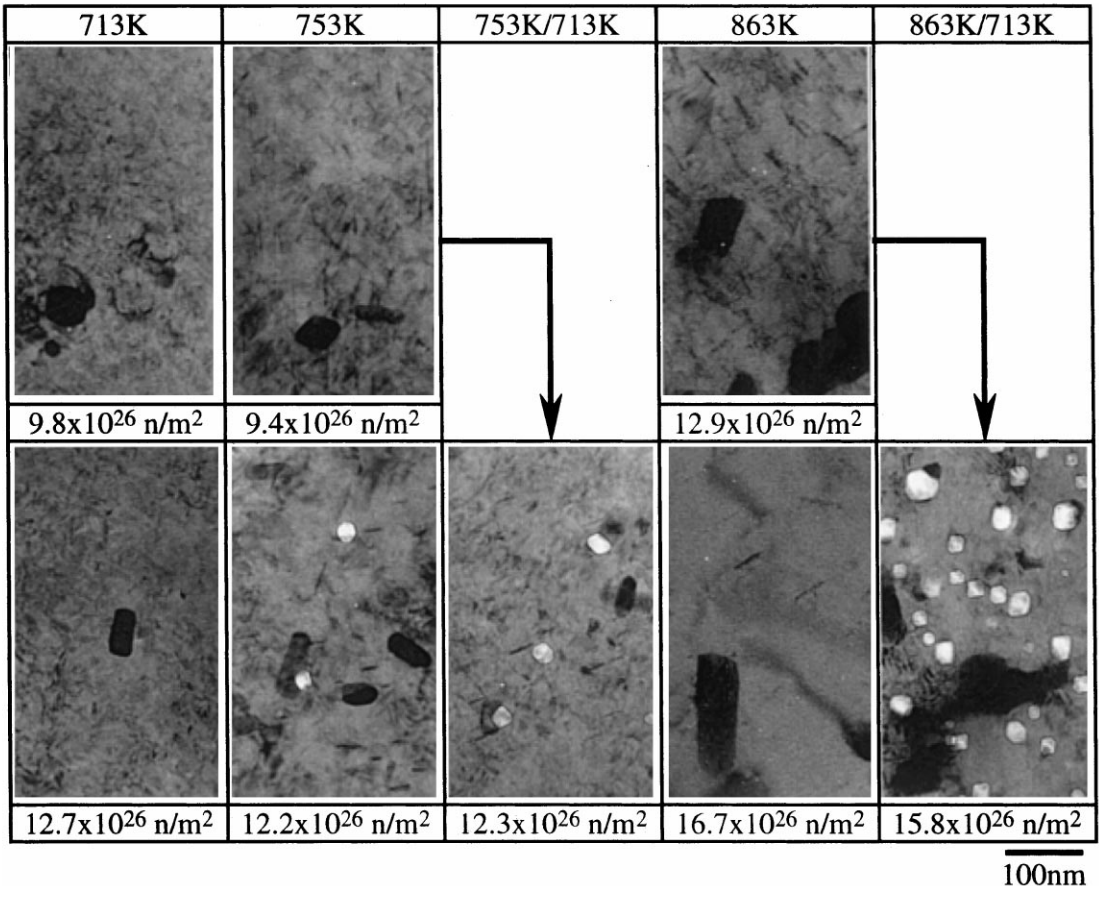

Fig. 1. Microstructure of P, Ti-modified 316 stainless steel in temperature change irradiation examination.

753 to 713 K. Precipitate formation in these temperature-changed specimens was more remarkable than that of the constant temperature irradiated specimen at 713 K. Phosphides cannot be observed in either temperature-changed specimens.

Fig. 2 shows microstructures of P, Ti-modified 316 stainless steels irradiated in the coupling irradiation program in Phenix and JOYO. At the end of Phenix irradiation period, void had formed a little in all specimens. Distribution of voids in the matrix was remarkably heterogeneous. Phosphides clearly formed in specimens irradiated at 793 and 843 K. But in the specimen irradiated at 753 K, phosphide was not observed. At the end of JOYO irradiation period, formation of voids is prominent in 753 K (Phenix)/773 K (CMIR) specimen. But, in other specimens, voids are slightly observed. In particular, void distribution of the specimen irradiated at 843 K (Phenix)/873 K (CMIR) is quite heterogeneous, and the void diameter ranges from 150 to 400 nm. As the fluence increases, precipitates were strongly formed. In 753 K (Phenix)/773 K (CMIR) and 793 K (Phenix)/843 K (CMIR) specimen, precipitates were mainly identified as M6C and G-phase. In 843 K (Phenix)/873 K (CMIR) specimen, precipitates were identified as M6C and Laves. Phosphides were observed in all specimen except for 753 K (Phenix)/773 K (CMIR) specimen. But, at 793 K (Phenix)/843 K (CMIR) specimen, phosphides tend to become indistinct as the fluence increases.

4. Discussion

4.1. Effects of irradiation temperature decreasing

As shown in results of step-wise temperature decreasing irradiation, void formation becomes prominent and phosphides, which have an inhibitory effect on void formation, are dissolved. It is thought that acceleration of void formation in temperature-decreased specimens is attributed to decreasing of critical radius of bubble and/or the dissolution of phosphide following by temperature decreasing.

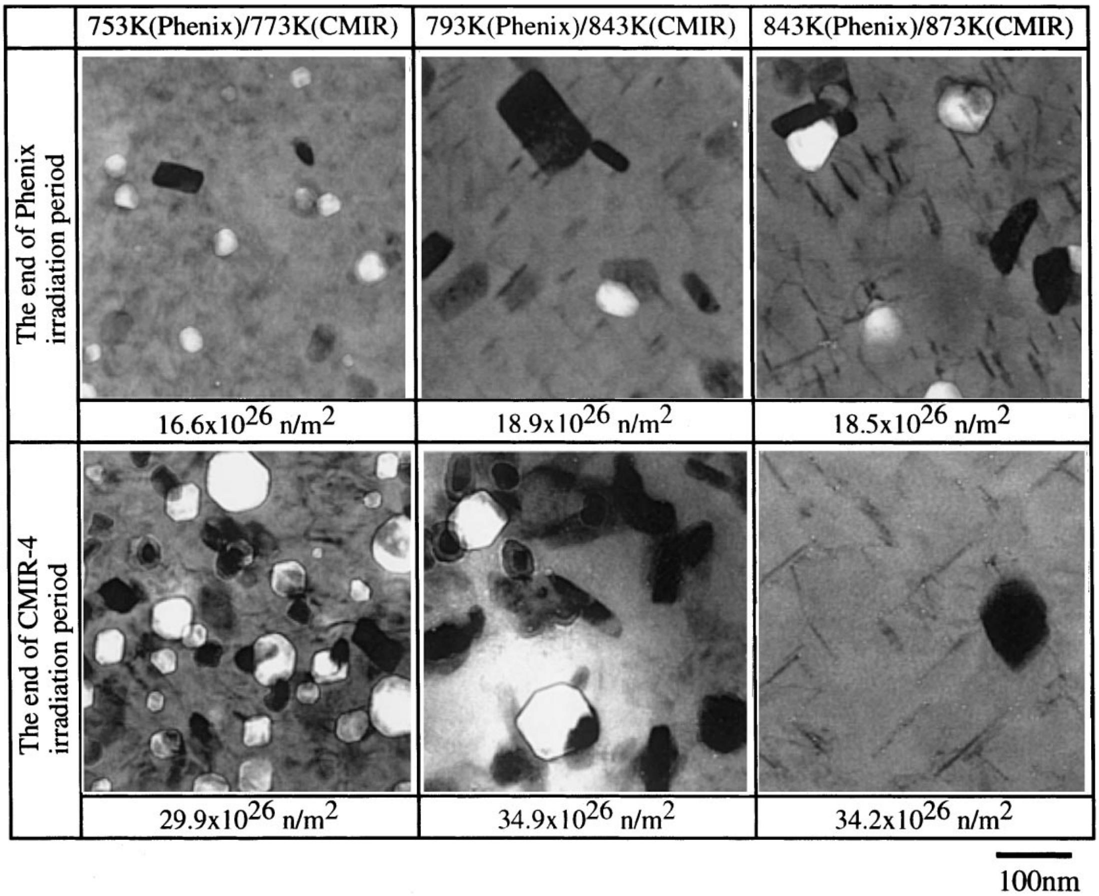

Fig. 2. Microstructure of P, Ti-modified 316 stainless steel in coupling irradiation examination.

Void formation is affected by vacancy supersaturation. Fig. 3 shows the dislocation density and the estimated value of vacancy supersaturation of temperature-decreased specimens. The vacancy supersaturation was estimated assuming that the only point defect sink is the dislocation. At the start of CMIR-4 irradiation period, dislocation density of temperature-decreased specimens is lower than that of specimen irradiated at 713 K, and vacancy supersaturation of these specimens increases stepwise. The critical radius of bubble should be decreased step-wise associated with this change of vacancy supersaturation. Therefore, for temperature decreased specimens, it is thought that bubble, which have stable radius corresponding to higher temperature, is possible to get over critical radius after a temperature decrease.

Concerning phosphide dissolution, it was reported that phosphide is a radiation induced precipitate and was formed only in the temperature range between 743 and 873 K [3]. The irradiation temperature of temperature-decreased specimens was changed from inside of formative temperature region for phosphide to outside. Stability of phosphide is controlled by balance between in-flow of phosphorous to phosphides and out-flow of that from them. The radiation induced segregation behavior should be changed remarkably with the change of vacancy supersaturation. Therefore, it is considered that dissolution of phosphides in the temperature-decreased specimens is caused by instability of phosphide due to dominant out-flow of phosphorous from the phosphide.

As mentioned above, it is well known that phosphide has inhibitory effect of void formation because of helium trapping effect at phosphide/matrix interfaces [3,4]. In associated with dissolved phosphide during irradiation period, it is thought that number of helium atoms in bubble is possible to get over critical number of helium atom, since release and redistribution of helium atom from phosphide/matrix interface to matrix should have occurred.

4.2. *Effects of irradiation temperature increasing*

As shown in results of Phenix/CMIR coupling irradiation, a few voids were observed at the end of Phenix irradiation period. But new voids is rarely formed in 793 K (Phenix)/843 K (CMIR) and 843 K (Phenix)/873 K (CMIR) specimens during CMIR irradiation period, as shown by the void diameter distribution in Fig. 4. This suppression of void formation may be caused by increasing irradiation temperature.

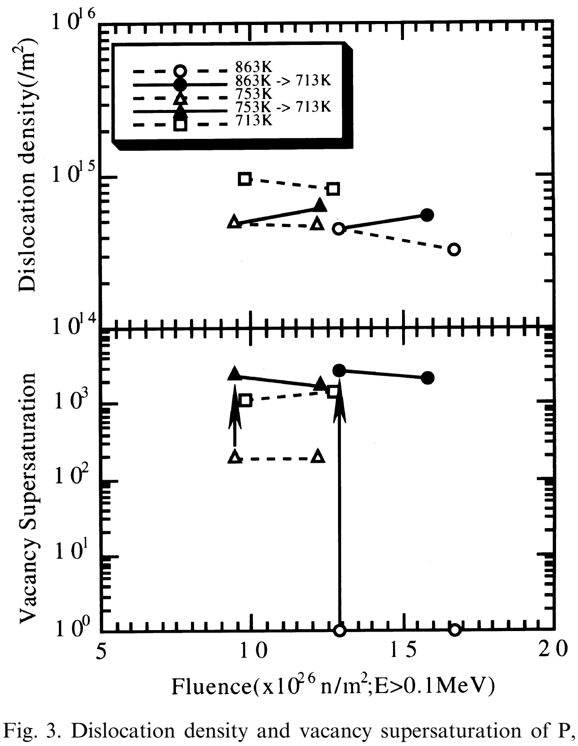

Fig. 3. Dislocation density and vacancy supersaturation of P, Ti-modified 316 stainless steel irradiated in step-wise temperature decreasing irradiation test.

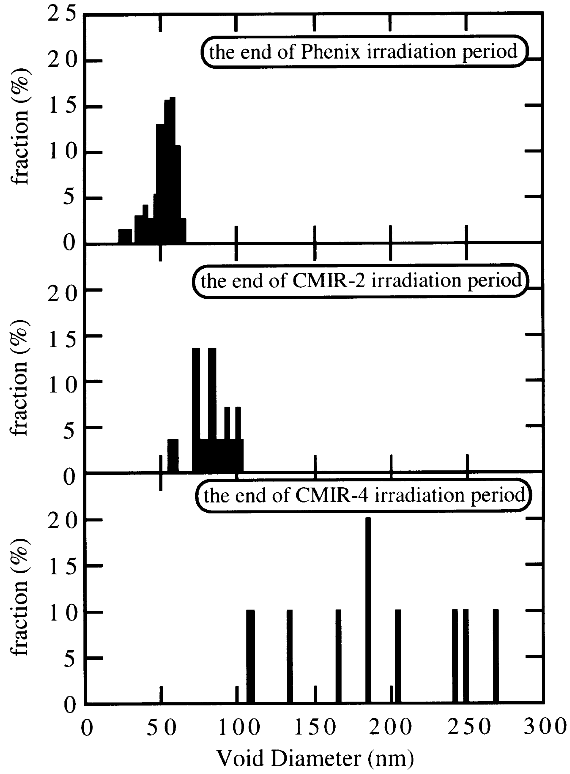

Fig. 4. Void diameter distribution on coupling irradiated P, Ti-modified 316 stainless steel at 843 K(Phenix)/873 K(CMIR).

Vacancy supersaturation should decrease and the critical radius of bubble should increase as irradiation temperature increases. Therefore, bubble-void conversion may be suppressed when the temperature is increased.

5. Conclusion

Based on the observation of microstructure change of neutron irradiated P, Ti-modified 316 stainless steels which experienced temperature change during irradiation, the influence of temperature change on void swelling was discussed.

With decreasing temperature, void formation is accelerated compared with that of constant temperature, while void formation is suppressed at the case of increasing temperature.

In these results, it is suggested that change of void formation behavior is caused by step-wise change of vacancy supersaturating associated with change of irradiation temperature and dislocation density.

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