MICROSTRUCTURAL EVOLUTION OF 9Cr-2WVTa STEELS IN HFIR-CTR-62/63 EXPERIMENT -

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OBJECTIVE

The objective of this work is to investigate microstructural evolution of nickel-doped and undoped ferritic/martensitic steels irradiated in HFIR-CTR-62/63 experiment in order to clarify the effect of helium generation rate on swelling and the change of the mechanical properties.

SUMMARY

The microstructures of reduced-activation ferritic/martensitic steels, 9Cr-2WVTa and 9Cr-2WVTa doped with 2% Ni, irradiated at 400°C up to 12 dpa in the High Flux Isotope Reactor (HFIR), were investigated by transmission electron microscopy. Specimens were tempered at two different temperatures in order to investigate the effects of tempering on microstructural evolution during irradiation. Before irradiation, the lath width of Ni-doped 9Cr-2WVTa was somewhat narrower and the dislocation density tended to be higher compared with 9Cr-2WVTa. Dislocation density of specimens tempered at 750°C was lower than that tempered at 700°C. In all steels, precipitates on grain and/or lath boundaries were mainly $M_{23}C_6$, and there were a few TaC along dislocations in the matrices. Irradiation-induced cavities were observed in all the steels. The cavity number density of the Ni-doped 9Cr-2WVTa was higher than that of 9Cr-2WVTa due to the higher concentration of helium; however, swelling in each steels was < 0.001% because cavity sizes were so small. There was no difference of cavity number density between the steels tempered at 700°C and 750°C, but the mean size of the cavities in the steels tempered at 750°C was larger than that tempered at 700°C. Irradiation-induced $\mathbf{a}_0$<100> and $(\mathbf{a}_0/2)$<111> type dislocation loops were observed in all steels; number density and mean diameter of $\mathbf{a}_0$<100> type loops was higher and larger than that of $(\mathbf{a}_0/2)$<111> type loops. There was a tendency for the number density of loops in Ni-doped 9Cr-2WVTa to be higher than that in 9Cr-2WVTa. In addition, the mean size of loops in the steels tempered at 750°C was larger than for those tempered at 700°C, while there was not much difference of number density between them. In the steels doped with Ni, irradiation-produced precipitates, identified as $M_6C(\eta)$-type carbide, were found in the matrices. Precipitation during irradiation might affect cavity and dislocation loop growth. In this experiment, the 9Cr-2VWTa-2Ni steel showed a larger increase of the transition temperature and a significant reduction in the upper shelf energy compared with the 9Cr-2WVTa steel. From these results, it might be suggested that the high density of cavities and precipitation in irradiated 9Cr-2VWTa-2Ni could affect Charpy properties, especially the reduction of upper shelf energy.

PROGRESS AND STATUS

1. Introduction

Ferritic/martensitic steels are attractive candidate structural first wall materials for fusion energy systems [1]. The high-energy neutrons produced by the D-T fusion reaction induce displacement damage and generate gas atoms in the materials from (n,p) and (n,α) reactions. It is considered positible that the simultaneous production of helium atoms from (n,α) reactions could strongly influence the nucleation of cavities. Therefore, to clarify the effect of helium atoms on the microstructural development and mechanical property change in martensitic steels under fast neutron irradiation, 9Cr-2WVTa and 9Cr-2WVTa with 2%Ni added were irradiated with neutrons in the High Flux Isotope Reactor (HFIR). In this experiment, specimens tempered at different temperature were prepared in order to investigate the effect of tempering on microstructural evolution during irradiation. Irradiation of Ni-doped steels in a mixed spectrum reactor like the HFIR results in the following transmutation reaction with the thermal neutrons: $^{58}Ni(n,\gamma)^{59}Ni(n,\alpha)^{56}Fe$, thus providing the possibility of studying the effects of the simultaneous production of displacement damage and helium production.

2. Experimental Procedure

9Cr-2WVTa and 9Cr-2WVTa-2Ni, normalized 0.5 h at 1050°C and tempered 1 h at 700°C (-A) or 750°C (-B), were prepared for this experiment; the compositions are given in Table 1. Standard 3-mm diameter transmission electron microscopy (TEM) disks were punched from 0.25-mm thick sheet stock. Irradiation was at 400°C in the HFIR-CTR-63 capsule in the High Flux Isotope Reactor (HFIR) to a neutron fluence of about 1.68×10^{22} n/cm^2 (E> 0.1 MeV) [2], resulting in a displacement dose of about 12 dpa. The details of the design, construction, and installation of HFIR-CTR-62/63 have been reported [3]. The irradiation conditions and the calculated helium concentrations in the steels are given in Table 2. TEM specimens were thinned using an automatic Tenupol electropolishing unit located in a shielded glove box. TEM disks were examined using a JEM-2000FX (LaB_6) transmission electron microscope. The foil thicknesses were measured by thickness fringes in order to evaluate quantitative defect density values.

3. Results and discussion

Before irradiation, the lath width of the 9Cr-2WVTa-2Ni steels was somewhat narrower and the dislocation density somewhat higher compared with the 9Cr-2WVTa steels. The dislocation density of specimens tempered at 750°C were lower than for the tempered at 700°C. In all steels, precipitates on grain boundaries were mainly $M_{23}C_6$, which were identified from the diffraction pattern, and there were a few TaC along dislocations in the matrices. The spacing of moiré fringes was used to identify the TaC. Figure 1 shows the unirradiated microstructures of the steels and the TaC carbides on dislocations.

Table 1. Chemical compositions of the specimens (wt%)

	9Cr-2WVTa	9Cr-2WVTa-2Ni
Cr	8.71	8.55
Ni	0.02	2.01
W	2.17	2.15
V	0.23	0.23
Ta	0.06	0.06
Mn	0.39	0.38
Al	0.021	0.021
Mo	< 0.01	< 0.01
Nb	< 0.01	< 0.01
Cu	< 0.001	< 0.001
Ti	< 0.01	< 0.01
C	0.098	0.098
P	0.014	0.014
S	0.003	0.003
Si	0.19	0.19
B	< 0.001	< 0.001
N	0.016	0.016
Fe	Bal.	Bal.

Table 2. Irradiation conditions and helium concentration

	dpa	appm He	He/dpa
9Cr-2WVTa	12.0	25	2.1
9Cr-2WVTa-2Ni	12.3	150	12.2

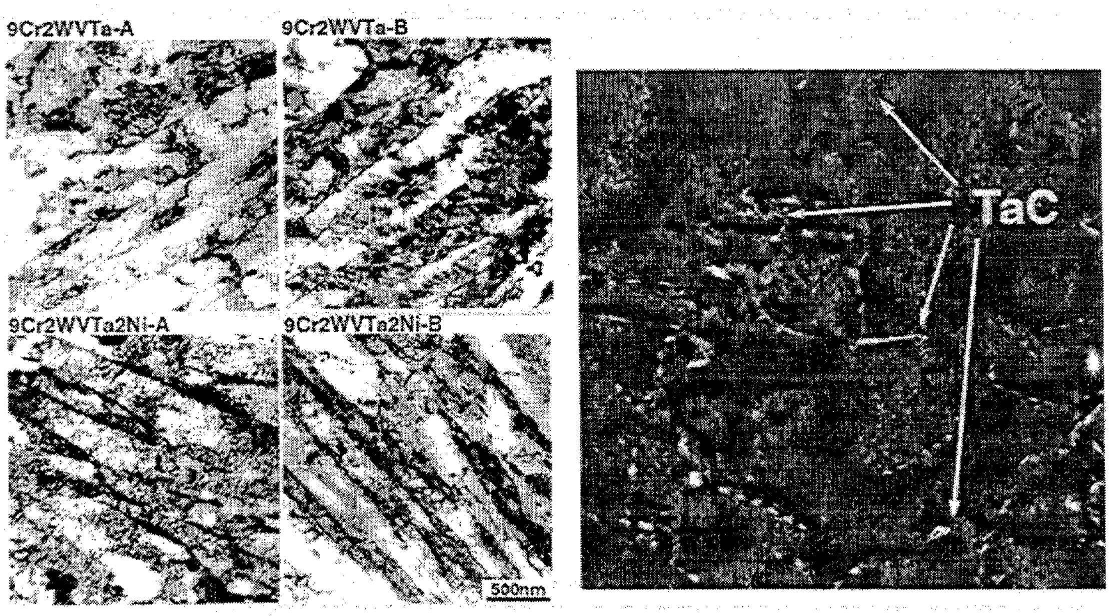

Fig.1. Microstructures of the unirradiated steels and TaC on dislocation.

3.1 Dislocations and dislocation loops

During irradiation of Fe-Cr binary alloys, dislocation evolution in an initially almost dislocation-free condition proceeds by the formation of interstitial-type dislocation loops with an $\mathbf{a}_0$<100> and/or ($\mathbf{a}_0$/2)<111> Burgers vector [4,5]. Figure 2 shows the dislocation segments and loops in the specimens after irradiation at 400°C to 12 dpa using the diffraction conditions: **g**=110, (**g**,4**g**). Table 3 summarizes the quantitative results of dislocation loops. The number density and the mean diameter of $\mathbf{a}_0$<100> type loops are higher and smaller than that of ($\mathbf{a}_0$/2)<111> type loops, respectively. In addition, there is a tendency for the number density of irradiation-induced ($\mathbf{a}_0$/2)<111> type dislocation loops in 9Cr-2WVTa-2Ni to be somewhat higher than those in the 9Cr-2WVTa. With respect to the two different tempering temperatures, the mean size of the loops in the steels tempered at 750°C was larger than for those tempered at 700°C, while there was little difference in the number density between them. Before irradiation, the dislocation density of specimens tempered at 700°C were higher than for those tempered at 750°C, and higher dislocation density tends to cause a smaller size of loops during irradiation. This suggests that the growth of irradiation-induced loops is controlled by heat treatment before irradiation.

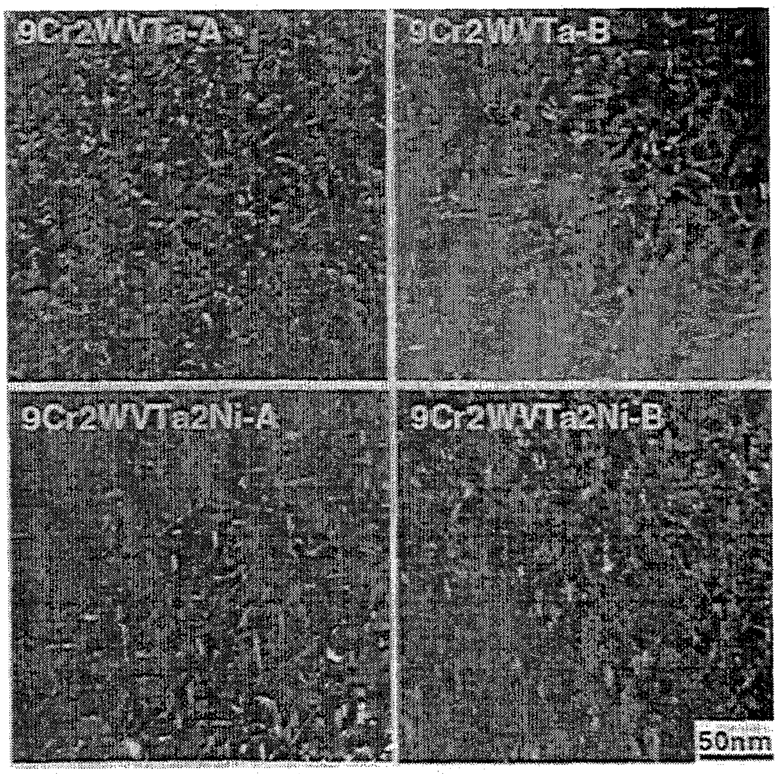

Figure 2. Dislocation segments and loops in 9Cr-2WVTa-A, 9Cr-2WVTa-B, 9Cr-2WVTa-2Ni-A and 9Cr-2WVTa-2Ni-B after irradiation at 400°C to 12 dpa using the diffraction conditions: **g**=110, (**g**,4**g**).

Table 3. Summary of dislocation structure before and after irradiation

Steel	a_0<100> type loops		$(a_0/2)$<111> type loops		Before irradiation
	Number density (m^{-3})	Mean diameter (nm)	Number density (m^{-3})	Mean diameter (nm)	Line density (m^{-2})
9Cr-2WVTa-A	$1x10^{22}$	18	$5x10^{21}$	28	$5x10^{14}$
9Cr-2WVTa-B	$1x10^{22}$	23	$4x10^{21}$	30	$2x10^{14}$
9Cr-2WVTa-2Ni-A	$1x10^{22}$	.17	$6x10^{21}$	16	$7x10^{14}$
9Cr-2WVTa-2Ni-B	$1x10^{22}$	23	$6x10^{21}$	28	$5x10^{14}$

3.2 Cavities

Figure 3 shows cavities of the specimens irradiated at 400°C to 12 dpa. Distribution of cavities is homogeneous in the matrices, and no cavities were observed on grain boundaries. Neutron irradiation induced tiny cavities with number densities of $3x10^{21}$ in 9Cr-2WVTa-2Ni-A, and -B. In 9Cr-2WVTa-A and -B, somewhat larger cavities were observed even though this steel contained very little helium compared with the Ni-doped steels. Swelling of each steel was estimated to be < 0.001%, suggesting the difference in helium concentrations did not affect swelling in this experiment, even though the cavity number densities correlated with the helium concentrations. Table 4 summarizes the quantitative results of the observations on the cavities.

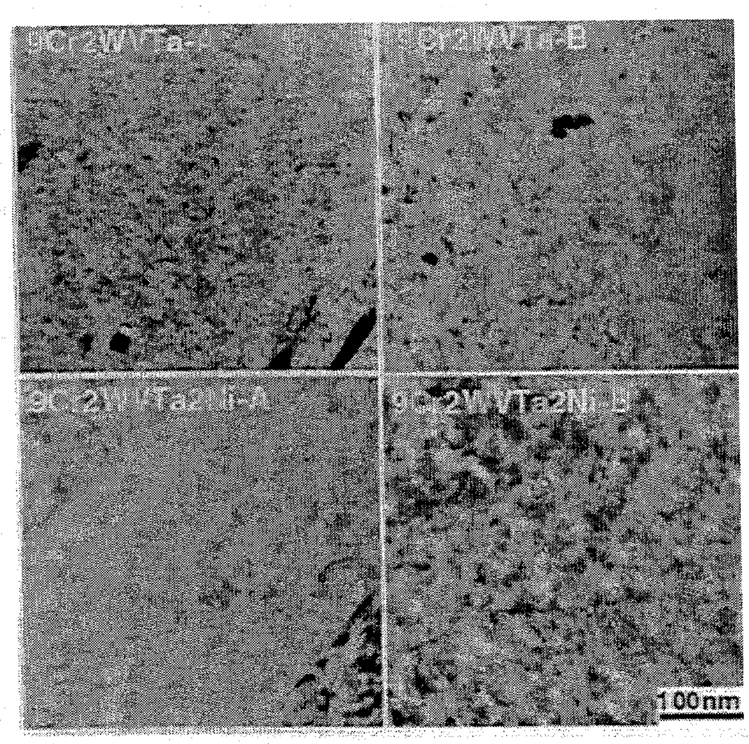

Figure 3. Cavities in 9Cr-2WVTa-A, 9Cr-2WVTa-B, 9Cr-2WVTa-2Ni-A and 9Cr-2WVTa-2Ni-B after irradiation at 400°C to 12 dpa.

Table 4. Summary of cavities formed during irradiation

Steel	Mean Size (nm)	Number Density (m^{-3})	Swelling (%)
9Cr-2WVTa-A	3	$1x10^{21}$	< 0.001
9Cr-2WVTa-B	9	$1x10^{21}$	< 0.001
9Cr-2WVTa-2Ni-A	3	$3x10^{21}$	< 0.001
9Cr-2WVTa-2Ni-B	4	$3x10^{21}$	0.001

3.3 Precipitates

Before irradiation, the alloys used in this study include a few TaC on dislocations and $M_{23}C_6$ on grain and/or lath boundaries, with the number density and the mean diameter of $<1x10^{20}$ m^{-3} and about 100 nm, respectively. There was little difference in $M_{23}C_6$ before and after irradiation in terms of size and distribution, but neutron irradiation did produce precipitates in the matrices of the Ni-doped steels. The spacing of moiré fringes was used to identify the irradiation-produced precipitates, which were identified as $M_6C(\eta)$-type carbide. Figures 4 shows the irradiation-induced $M_6C(\eta)$-type carbides in the 9Cr-2WVTa-2Ni-A and –B steels after irradiation at 400°C to 12 dpa. Precipitation of a Cr- and Ni-rich $M_6C(\eta)$ has been observed in a number of 8-12Cr steels that contain >0.3wt% Ni during FBR (Fast Breeder Reactor) and HFIR irradiation at about 400°C [6-9]. Table 5 summarizes the quantitative results of precipitates.

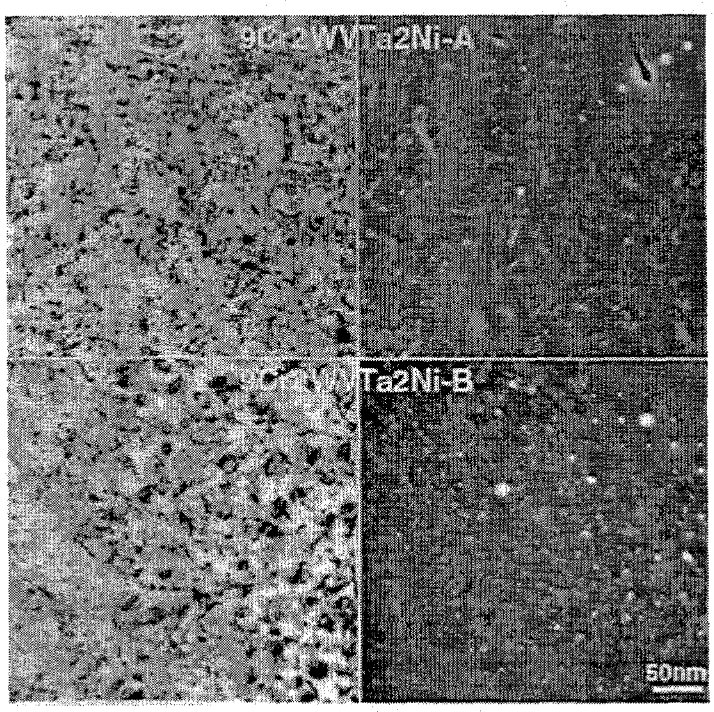

Figure 4. Irradiation-induced precipitates in 9Cr-2VWTa-2Ni-A and 9Cr-2VWTa-2Ni-B after irradiation at 400°C to 12 dpa.

Table 5. Summary of precipitates in the steels after irradiation at 400°C

Steel	$M_{23}C_6$		$M_6C(\eta)$	
	Mean Size (nm)	Number Density (m^{-3})	Mean Size (nm)	Number Density (m^{-3})
9Cr-2VWTa-A	95	$<1x10^{20}$	-	-
9Cr-2VWTa-B	102	$<1x10^{20}$	-	-
9Cr-2VWTa-2Ni-A	92	$<1x10^{20}$	7	$2x10^{21}$
9Cr-2VWTa-2Ni-B	98	$<1x10^{20}$	6	$2x10^{21}$

The Charpy specimens from HFIR-CTR-62/63 experiment showed some differences between 9Cr-2VWTa and 9Cr-2VWTa-2Ni steel [10]. Table 6 and 7 summarize the Charpy properties and the tensile properties of the specimens, respectively. The 9Cr-2VWTa-2Ni steel showed larger increase of the strength and the transition temperature and a significant reduction in the upper-shelf energy compared with 9Cr-2WVTa steel. As mentioned above, precipitation of $M_6C(\eta)$-type carbide and a higher number density of cavities were observed in the irradiated 9Cr-2VWTa-2Ni steel. From these results, it might be suggested that a high density of cavities and precipitation in the irradiated 9Cr-2VWTa-2Ni could affect the yield and tensile strength and Charpy properties.

Table 6. Charpy properties of steels in HFIR-CTR-62/63 experiment [10].

Steel	Irrad. Temp. (°C)	Trans. Temp. (°C)	ΔDBTT (°C)	Upper-Shelf Energy (J)
9Cr-2VWTa	Unirrad.	-94		10.8
	400	-15	79	6.5
9Cr-2VWTa-2Ni	Unirrad.	-113		10.8
	400	21	133	n/m

Table 7. Tensile properties of steels in HFIR-CTR-62/63 experiment [10].

Steel	Irrad. Temp.	Test Temp.	Strength (MPa)		Elongation (%)	
	(°C)	(°C)	Yield	Tensile	Uniform	Total
9Cr-2VWTa	Unirrad.	400	715	817	1.6	4.5
	400	400	963	983	0.6	5.8
9Cr-2VWTa-2Ni	Unirrad.	400	733	824	1.6	4.3
	400	400	1034	1075	0.6	5.7

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