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Effect of combined addition of Ti and P on creep rupture properties of helium implanted Fe–25%Ni–15%Cr alloy

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Abstract

Creep rupture tests after hot helium implantation (60–70 appm) at 923 K were conducted on thermomechanically treated Fe–25%Ni–15%Cr alloys with and without combined addition of Ti and P. The results obtained were compared with those of single additions in order to gain systematic information concerning the effects of Ti and/or P modifications on the austenite matrix. In terms of creep rupture time Ti-bearing alloys could withstand deleterious helium effects. In contrast, the alloy of no addition suffered from the most pronounced degradation by helium. On the other hand, considerable decrease in rupture elongation was discerned in all alloys after the implantation though the degree of embrittlement varied from alloy to alloy. The largest degradation was again observed for the alloy with no addition. These results suggest that helium induced mechanical degradation of austenitic alloys could be suppressed by proper additions of above mentioned elements and appropriate microstructure arrangements.

1. Introduction

Material degradation caused by heavy irradiation of 14 MeV neutrons has been considered to be one of the most crucial problems to be solved for the construction of future fusion reactors. Many investigations of every stripe have been hence carried out throughout these several decades, so as to contribute to the development of radiation resistant high performance materials. As a result of such research activities, it is now clarified that minor additions of Ti and/or P to Fe–Ni–Cr austenitic materials, which are candidates for first wall/blanket structural components, tend to suppress radiation induced void swelling and creep of these materials. Since the influence of those elements on another harmful radiation effect, i.e. helium embrittlement, is also a major concern, investigations have been done by means of fission reactor irradiation and accelerator implantation of helium etc. (see, e.g. Refs. [1–10]).

Along with these activities we have performed systematic studies on the effect of those addition to model Fe–Ni–Cr ternary austenitic alloys using creep testing after hot helium implantation, in order to obtain further fundamental knowledge and understanding about the influence of Ti and P modifications on helium induced degradation of mechanical properties. In this article, effects of combined addition will be reported and compared with those of individual additions examined before [11].

2. Experimental

The materials used are model Fe–25%Ni–15%Cr austenitic alloys with and without a combined addition of Ti and P. The results of chemical analysis on them are shown in Table 1, together with those of alloys with single additions which were previously investigated [11]. The 17 kg ingot of each alloy was prepared from high purity virgin raw materials by vacuum induction melting. The 0.17 mm thick sheets were fabricated from the ingots through ordinary hot working and cold rolling processes. All heat treatments in these processes were executed at 1513 K in a vacuum and followed by Ar gas quenching with an average cooling rate of about 30–20 deg./s. The unusually high temperature anneals and

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Table 1
Chemical composition of alloys investigated (wt%)

Alloy	C	N	O	Ni	Cr	Ti	P	S	Fe
7807 (None)	0.065	0.002	0.034	25.85	15.17	–	0.002	0.003	Bal.
8801 (+Ti)	0.071	0.001	0.007	23.63	14.88	0.29	<0.002	<0.003	Bal.
8802 (+P)	0.071	0.001	0.006	22.58	14.88	–	0.052	<0.003	Bal.
8803 (+Ti,P)	0.071	0.001	0.006	23.63	14.76	0.27	0.052	<0.003	Bal.

rapid cooling were adopted to eliminate unintended precipitation [7,12] before preimplantation treatments described below, in which precipitation is expected to take place under controlled condition, particularly in case of materials containing MC type precipitate formers. The conducted preimplantation treatments are documented in Table 2, together with resultant material microstructures. The temperature and time of the first treatment, a grain size control anneal, were selected to yield a mean grain size between 10 and 20 μm, since it is established that more than several grains are necessary through every direction in a gauge section to assess mechanical properties of bulk materials with thin specimens (see, for instance, Refs. [13–15]). Conditions of the subsequent thermomechanical treatments were decided by referring the result of disk-bend tests on HFIR irradiated US-PCA [4]. The precipitates documented in Table 2 should have been formed during aging. Among them, MC particles, which is called as "secondary" MC [16,17], oriented at a cube-on-cube relation to the matrix. In the cases of Ti-modified alloys, the other version of MC besides those in Table 2 was sparsely distributed with a size of hundreds nm at a random orientation to the matrix. They are so-called "primary" MC [16,17] and most probably arose during grain size control annealing. Specimens for creep testing, the gauge section of which was 10 mm long, 4 mm wide and 0.15 mm thick, were die-stamped out after the final aging.

Helium implantation was carried out at 923 K to a concentration of about 60–70 appm with an implantation rate of $1–2 \times 10^{-3}$ appm/s, using a 26 MeV helium-3 ion beam from a cyclotron. The energy of incident ions was adjusted by an rotating energy degrader, which comprises 32 Al-foils with different thicknesses, for the purpose of uniform helium distribution all though the specimen depth. In addition, the beam was scanned over the irradiation area with two directions. The temperature fluctuation during irradiation was compensated by an infra-red lamp heater with rapid response.

Creep rupture tests after implantation were run on at the same temperature in a vacuum with conventional direct loading type machines. The deviation from the stated temperature during creep tests was less than ±1 deg. Such creep rupture tests were similarly done for the reference control specimens which were not implanted with helium but had exactly the same metallurgical histories. After creep tests all the ruptured samples were fractographically inspected with a scanning electron microscope (SEM). In addition, helium bubble microstructures in Ti and P modified alloy were observed under a transmission electron microscope (TEM).

The experimental details are presented elsewhere [18].

3. Results and discussion

Fig. 1 gives creep stress versus time to rupture relations for both sample materials in helium implanted and unimplanted conditions. The earlier results on alloys of single additions are also shown in the figure for comparison. In the absence of helium, Ti-bearing alloys exhibited higher strengths than the others. This behavior probably can be explained by finer precipitation of intragranular carbides in the former materials (see,

Table 2
Preimplantation treatments and resultant microstructures

Alloy	Preimplantation treatment [a]	Grain size (μm)	Precipitate			
			Sort	Size		Number density
				Matrix (nm)	G.B. (nm)	Matrix (m^{-3})
7807 (none)	GCA(1273 K, 3.6 ks) + A(1073 K, 36 ks) +CW(10%) + A(1023 K, 18 ks)	15.2 ± 1.0	$M_{23}C_6$	100–500	100–1000	$\approx 1 \times 10^{18}$
8801 (+Ti)	GCA(1323 K, 3.6 ks) + A(1073 K, 36 ks) +CW(10%) + A(1023 K, 18 ks)	17.3 ± 1.1	MC $M_{23}C_6$	20–150	7–50 100–1000	$\approx 2 \times 10^{19}$
8802 (+P)	GCA(1253 K, 3.6 ks) + A(1073 K, 36 ks) +CW(10%) + A(1023 K, 18 ks)	14.3 ± 0.8	$M_{23}C_6$	50–700	100–1000	$\approx 2 \times 10^{18}$
8803 (+Ti,P)	GCA(1323 K, 3.6 ks) + A(1073 K, 36 ks) +CW(10%) + A(1023 K, 18 ks)	15.0 ± 1.2	MC $M_{23}C_6$	20–150	7–50 100–1000	$\approx 2 \times 10^{19}$

[a] GCA: grain size control annealing; A: aging; CW: cold work.

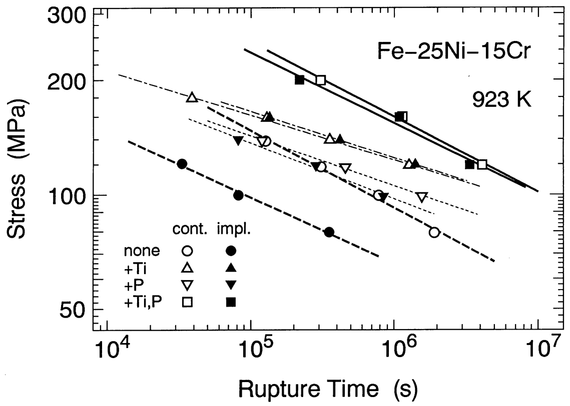

Fig. 1. Creep rupture strength as a function of creep life at 923 K for thermomechanically treated Fe–25%Ni–15%Cr alloys with and without Ti, P modifications in helium implanted ($C_{He}=60$–70 appm) and unimplanted conditions.

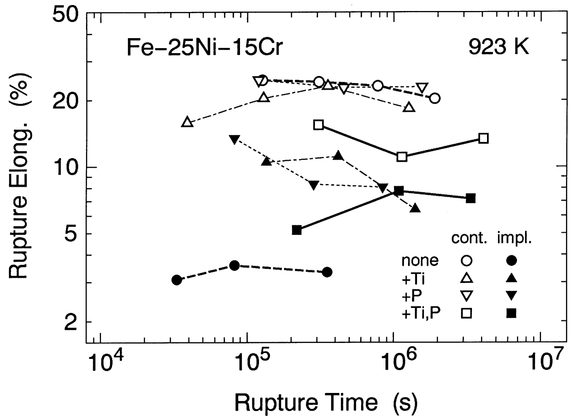

Fig. 2. Creep rupture elongation as a function of creep life at 923 K for thermomechanically treated Fe–25%Ni–15%Cr alloys with and without Ti, P modifications in helium implanted ($C_{He}=60$–70 appm) and unimplanted conditions.

Table 2). The larger strengthening of the alloy with combined addition in comparison with merely Ti-added one may be associated with solution hardening of P [19] and/or precipitation hardening by fine M_2P, which was formerly suggested as a cause of improved creep strength by P addition in Ti-modified austenitic stainless steels [20,21]. In Ti-containing alloys, helium implantation seems to cause no significant degradation and no notable influence of P modification on the alloy of Ti single addition was observed concerning helium effects on creep strength. On the other hand, helium brought about a considerable decrease in terms of creep rupture time for alloys without Ti addition. The extent of the reduction was around 60% and almost reached to 90% for P-modified alloy and alloy without modification, respectively. This result proves that combination of Ti addition and appropriate microstructure modification through thermomechanical treatments could improve resistance to mechanical deterioration by helium regardless of the presence or absence of P.

All sets of rupture time–stress data in Fig. 1 fitted well with usual linear regression. Table 3 presents stress exponents n of the best fits of creep power law, $t_r \propto \sigma^{-n}$, for each test series. As described in the table, the stress exponents of implanted and unimplanted specimens are very similar for every alloy. This fact suggests that creep constraint growth would be the rupture time controlling mechanism [22].

Table 3
Stress exponents n of creep power law fitting, $t_r \propto \sigma^{-n}$, for helium implanted and unimplanted specimens tested at 923 K

Alloy	No He	60–70 appmHe
7807 (None)	4.9	5.7
8801 (+Ti)	8.5	8.2
8802 (+P)	7.6	6.8
8803 (+Ti,P)	5.0	5.2

Fig. 2 presents the relation between creep rupture elongation and rupture time. The lowest values of Ti and P added alloy in the helium free condition may reflect the highest rupture strength of it. Different from what was perceived for rupture strength, substantial decrease of rupture elongation by helium was discerned in all materials. Though the degree of helium embrittlement was almost an order of magnitude high for the alloy not modified with Ti and/or P, the rupture elongations of implanted samples of other alloys stayed roughly between 40–70% of unimplanted values, showing no meaningful effect of P addition on Ti single modification. The alloy with no modification hence suffered the most severe deterioration by helium in terms of embrittlement, as well.

Results of fractographical investigations are exemplified in Fig. 3. For all alloys examined, including those with single additions, grain boundary fracture was frequently observed for helium implanted specimens, although helium free controls failed predominantly in a transgranularly ductile fashion. Fig. 4 reveals this feature by plotting the percentage of grain boundary decohesion against rupture time in both helium free and implanted conditions. As can be seen in the figure, helium explicitly promoted intergranular separation and the tendency was generally intensified with increasing rupture time in nearly the same manner previously reported [23,24], except for the material with no Ti and P addition. In this alloy fairly large values beyond 90%, which probably mirror larger degradation by helium, were already perceived in short rupture times.

Helium implanted and creep ruptured samples of the alloy of combined addition were examined by TEM with special attention for helium bubble microstructure

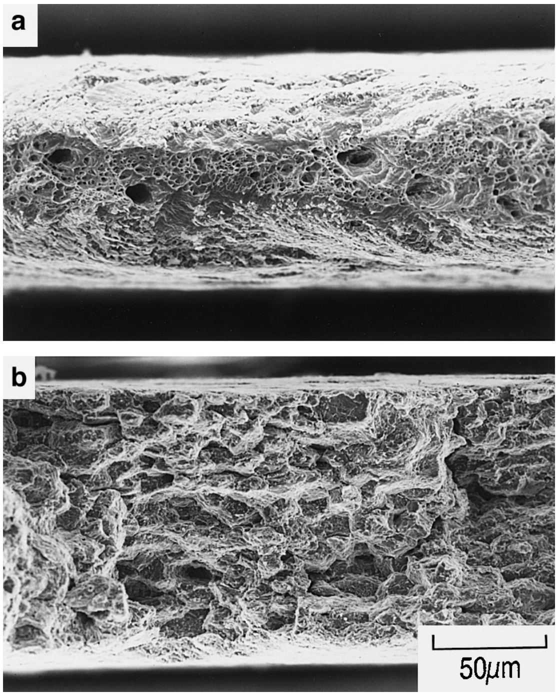

Fig. 3. SEM fractographs showing an enhancement of grain boundary decohesion by helium in creep rupture (Photos were taken on Alloy 8803 (+Ti, P) tested at 923 K, 160 MPa); (a) unimplanted control ($t_r = 1.14$ Ms); (b) helium implanted specimen ($C_{He} = 60$ appm, $t_r = 1.09$ Ms).

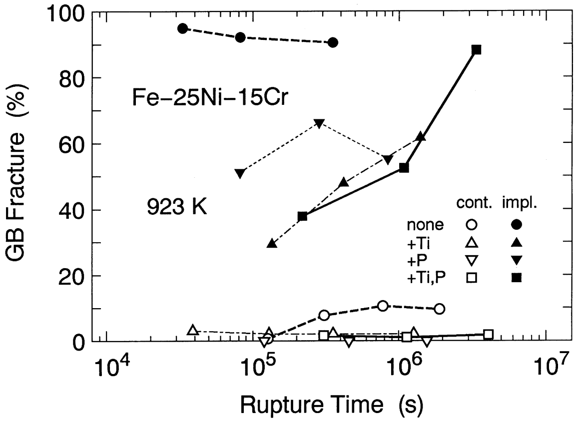

Fig. 4. Percentage of intergranularly fractured areas on rupture surface as a function of creep life at 923 K for helium implanted ($C_{He} = 60$–70 appm) specimens and unimplanted controls of thermomechanically treated Fe–25%Ni–15%Cr alloys with and without Ti, P modifications.

because it often has some correlations to helium effects on mechanical properties. Fig. 5 shows general appearances of bubbles both in the matrix and at grain boundaries. As illustrated in the figure, very tiny bubbles of

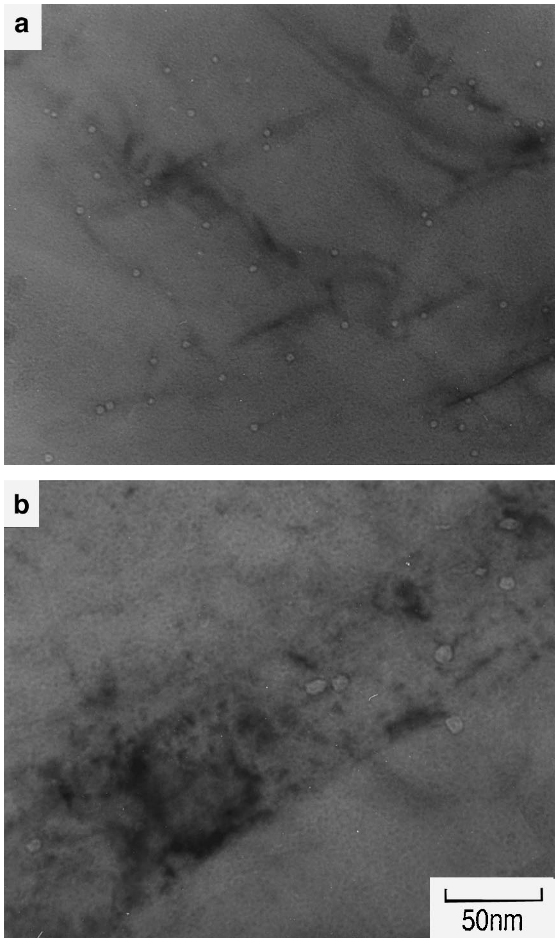

Fig. 5. TEM micrographs of helium bubble microstructures in thermomechanically treated Alloy 8803 (+Ti, P) after post helium implantation creep rupture ($T = 923$ K, $C_{He} = 60$ appm, $\sigma = 160$ MPa, $t_r = 1.09$ Ms); (a) in the matrix; (b) at a grain boundary.

approximately 2–4 nm in size were distributed all over the grain interiors at a number density around 5×10^{21} m^{-3} and, similar but slightly larger bubbles were also observed at grain boundaries. Some bubbles in the matrix were preferentially situated at the interfaces of MC precipitates as shown in Fig. 6. The presence of these bubble-precipitate associations would impede helium motions from the inside of grains to the grain boundaries and eventually may contribute to lessen detrimental helium effects on mechanical properties.

4. Conclusions

Helium effects on mechanical properties of Fe–25%Ni–15%Cr alloys with and without a combined minor addition of Ti (0.3%) and P (0.05%) were

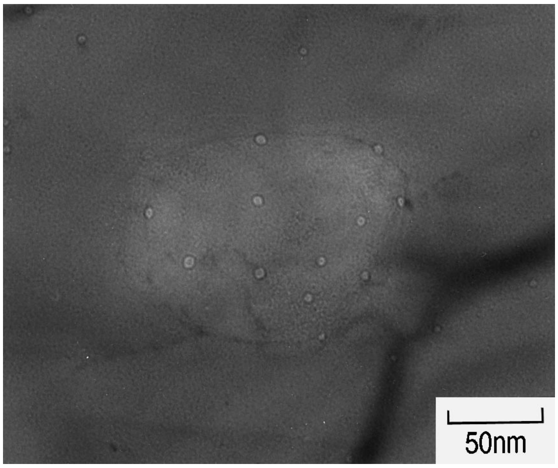

Fig. 6. TEM micrograph showing preferential helium bubble nucleation at an intragranular MC particle in a helium implanted and creep-ruptured specimen ($T = 923$ K, $C_{He} = 70$ appm, $\sigma = 200$ MPa, $t_r = 219$ ks) of thermomechanically treated Alloy 8803 (+Ti, P).

investigated through creep testing after hot helium implantation ($T = 923$ K, $C_{He} = 60$–70 appm) and, were compared with those of formerly tested ones of single modifications. The main results obtained are as follows.

1. The creep life of the alloy containing both Ti and P was not degraded by helium, similarly to the case of single Ti modification. The reason for this good resistance against the detrimental helium effect is, at least, partially due to preferential capturing of helium bubbles at intragranular MC precipitates in these alloys. This fact suggests that Ti modification and appropriate controlling of precipitate microstructure may help to suppress helium induced mechanical deterioration even in alloys with the intermediate nickel content.
2. The alloy not bearing Ti and/or P exhibited the largest degradation by helium, mostly reaching to the reduction by one order of magnitude in terms of both creep rupture strength and elongation. It is therefore concluded in combination with the result documented in (1) that the P addition of the above mentioned level seems to cause no harmful influence on helium embrittlement response, irrespective of whether Ti is simultaneously added or not.

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