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CREEP OF THE NOVEL AUSTENITIC HEAT RESISTANT STEEL OF FE-20CR-30NI-2NB UNDER STEAM ATMOSPHERE AT 1073 K

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ABSTRACT

In this study, we have examined the creep of a novel austenitic heat resistant steel of Fe-20Cr-30Ni-2Nb (at.%) steel at 1073K in steam and air atmospheres. Our studied steels were Fe-20Cr-30Ni-2Nb (base steel) and that with 0.03 at. %B (B-doped steel) . The addition of boron is to intentionally increase the area fraction of Laves phase on grain boundaries (ρ). The specimen with ρ = 43% (base steel pre-aged at 1073 K/240 h) exhibits the rupture life of 262 h, whereas the rupture life of the specimen with higher ρ of 80% (B-doped steel pre-aged at 1073 K/240 h) is 833h, which is about three times longer than that of the specimen with ρ = 43%. The specimen with ρ = 80% exhibits smaller creep rate than those with lower ρ than 43% in the entire creep stage. In addition, all specimens show the creep rupture strain of about 60%. The creep rupture life is almost same to that tested under air, whereas the creep rupture strain is slightly smaller (a few percent) than that under air. In the surface of the creep ruptured specimen in steam, the intergranular oxides associated with voids or cavities are often present and grow along grain boundaries to over 100 μm in depth. The intergranular oxidation occurs more extensively in steam rather than air. These results demonstrate that stable Fe_2Nb Laves phase on grain boundary could increase the creep resistance of the present steel at 1073K without ductility loss in steam as well as air, resulting in the pronounced extension of rupture life. The intergranular oxidation accelerated by steam would not give a serious effect on the creep properties of the present steel below 10^3 hours in rupture life.

INTRODUCTION

The advanced ultra-super critical (A-USC) thermal power plants operated under steam condition of 973 K/ 35 MPa is necessary to develop materials with 10^5 h creep rupture strength more than 100 MPa at 973 K [1-4]. Ni-base superalloys can meet the condition [4], but due to the high costs, austenitic heat resistant steels are receiving attention. However, the conventional austenitic heat resistant steels strengthened by carbides are not strong enough, due to microstructure instability during exposure at elevated temperature. Takeyama *et al.* [5-7] have pointed out to develop a new austenitic heat-resistant steels strengthened by thermally equilibrium intermetallic phases, and studied Fe_2M Laves phase in equilibrium with γ phase in the Fe-Ni-M ternary systems at 1473 K, where M is transition metals. Base on the results, we have developed a novel austenitic heat resistant steel of the Fe-20Cr-30Ni-2Nb (at.%) strengthened by TCP (Topologically Close-Packed) Fe_2Nb Laves phase on grain boundary, together with GCP (Geometrically Close-Packed) Ni_3Nb phase in grain interior [8]. This steel exhibits much higher creep rupture strength [9-11] than the conventional austenitic heat resistant steels strengthened by carbides. Its creep rupture strength can meet the requirement for A-USC power plant (973 K, 100 MPa, 10^5 h). This superior long-term creep rupture strength would be mainly due to the stable TCP Laves phase suppressing local deformation around grain boundaries, which is so-called *"grain boundary precipitation* *strengthening*" (GBPS) mechanism [10,11]. And the sufficient oxidation resistance to sustain the creep strength under severe environment of steam condition is also needed to realize the A-USC power plant. It was found that the Fe-20Cr-30Ni-2Nb steel exhibits superior steam oxidation resistance to the existing steels at 973 K [12], whereas the steam oxidation resistance under constant stress and its effect on the creep strength, in particular GBPS mechanism by TCP Laves phase should be important issues.

In this study, in order to indentify whether the grain boundary TCP Laves phase can increase the creep rupture strength at 1073 K under steam atmosphere, we have examined creep properties of the present steels with different area fractions of TCP Laves phase on grain boundary (ρ) at 1073 K under air and steam atmospheres.

EXPERIMENTAL PROCEDURES

The chemical composition of our studied steels were Fe-20Cr-30Ni-2Nb at.% and that with 0.03 at.% B. Hereafter, the former and the latter are expressed by base steel and B-doped steel, respectively. The addition of boron is to intentionally increase the area fraction of Laves phase on grain boundaries (ρ), based on our previous result [9]. These steels were prepared by vacuum induction melting and hot-forged into rods with a diameter of 12 mm at 1553 K. Subsequently, they were solution treated at 1473-1523 K/2 h to adjust the grain size to about 150 μm. The solution treated steels were pre-aged at 1073 K to control ρ. Their microstructures were observed by using a scanning electron microscope (SEM). We have measured the length of grain boundaries covered by Laves phase particles and the total length of grain boundaries on the mechanically polished samples, which can give a quantitative value of ρ. The total length of measured grain boundaries was more than 1000 μm for each specimen. The creep tests were conducted at 1073 K /70 MPa in air and steam atmospheres. In particular, creep test in steam atmosphere was conducted by using our originally designed creep test machine. Its schematic illustration is shown in **Fig. 1**. All components of extensometers, waterproof differential liner valuable transducers and a creep test specimen were shielded by a quartz tube chamber, which enables us to measure the precise creep elongation under steam atmosphere. Water was pumped up at 4.0 x 10^{-3} *l* / min and then changed to steam by pre-heaters, followed by steam flowing into

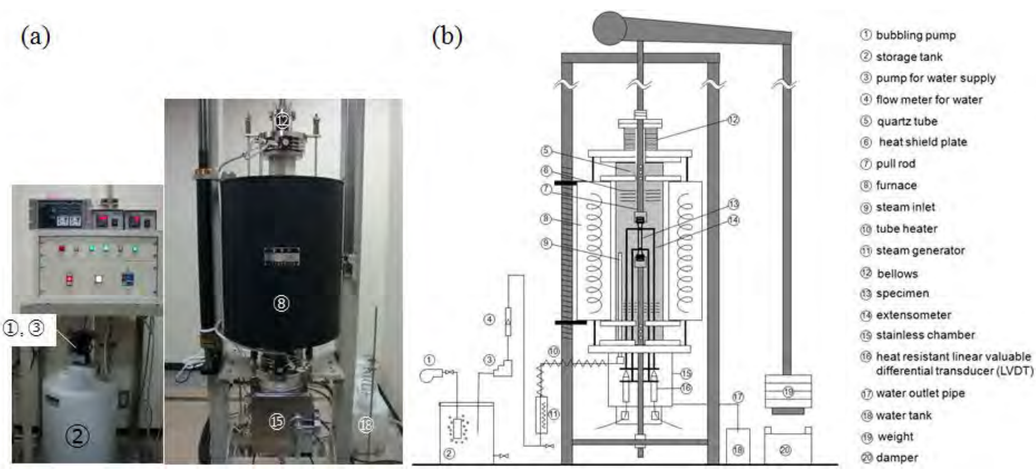

Figure 1: (a) Appearance of the creep test machine used in this study and (b) its schematic illustration.

the chamber. Note that bubbling water in the storage tank can keep a constant amount of dissolved oxygen in flowing water. We have observed microstructures of different microscopically strained regions in the gage portion of creep ruptured specimens. The macroscopic strain was determined by measuring the area reduction for the gage portion of the ruptured specimens.

RESULTS

Microstructure prior to Creep Test

In order to control the area fraction of grain boundary Laves phase (ρ), the steels were intentionally aged at 1073 K, where only the Fe_2Nb Laves phase precipitates [8, 9]. **Figure 2** shows back scattering electron images (BEIs) showing microstructures of the base and B-doped steels aged at 1073 K for 240 h and 1200 h. In the base steel aged for 240 h, there are precipitates of Fe_2Nb Laves phase with a length of about 1 μm on grain boundaries (ρ = 43%). In grain interior, the plate-shaped Laves phase with a length of about 1 μm precipitates homogenously. After 1200 h, the precipitates of Laves phase cover a half area of grain boundaries (ρ = 52%), whereas the precipitation morphology of Laves phase within grains slightly changes. In B-doped steel aged for 240 h, a large part of grain boundaries is covered by precipitates of Laves phase (ρ = 80%). ρ increases to 89% after 1200 h. The morphology of the Laves phase precipitated in grain interior is coarser than that observed in the base steel. Its precipitation density becomes lower after 1200 h aging.

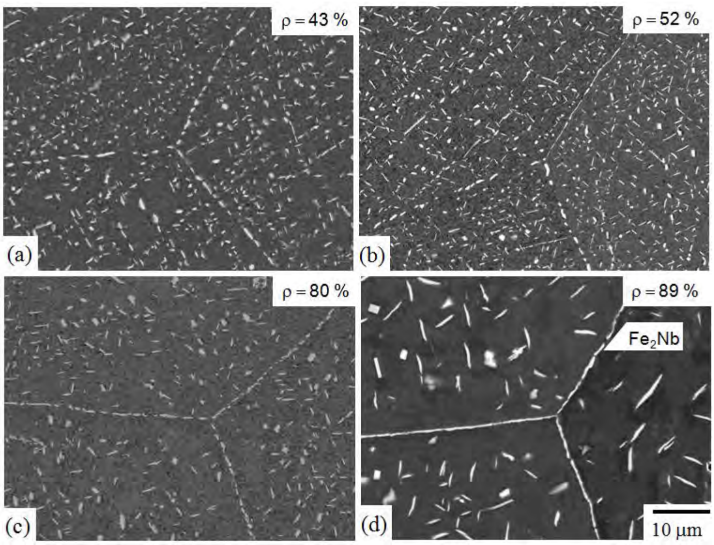

Figure 2: Microstructures of (a, b) base and (c, d) B-doped steels aged at 1073 K for (a, c) 240 h and (b, d) 1200 h.

Creep Properties

Figure 3 shows (a) creep rate/time curves and (b) creep rate/strain curves of the solution treated base and B-doped steels at 1073 K/70 MPa in air and steam atmospheres. Base steel exhibits a minimum creep rate of 1.1 x10^{-3}, which is independent of the atmosphere. Its rupture life (t_r) and rupture strain (ρ) under air are almost same to those under steam atmosphere. In B-doped steel, the creep rate decreases to about 2 x 10^{-4} /h after loading till 1 h, but increases and then keeps constant creep rates of 8.5 x10^{-4} in air and 5.0 x10^{-4} h in steam atmosphere, followed by creep acceleration. t_r under air (293 h) is lower than t_r under steam (376 h) ,whereas ε_r under air (0.31) is higher than ε_r under steam (0.25).

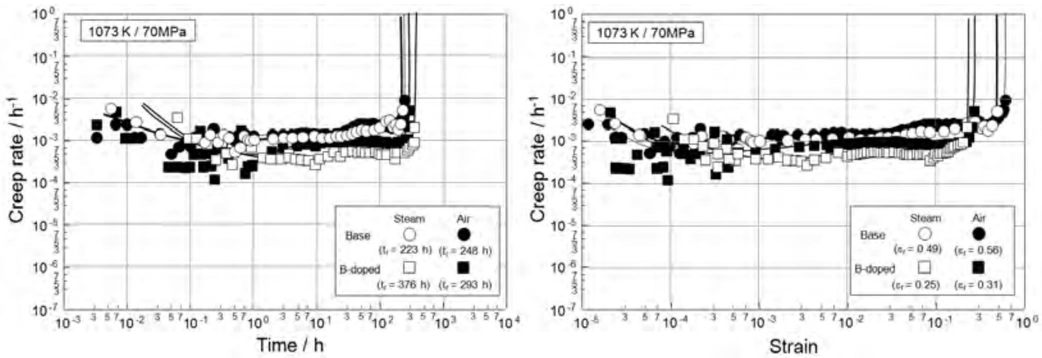

Figure 3: (a) Creep rate/time curves and (b) creep rate/strain curves of base and B-doped steels (as solution treated) at 1073K/70 MPa.

Figure 4 shows (a) creep rate/time curves and (b) creep rate/ strain curves of the pre-aged (1073 K/240 h) base and B-doped steels at 1073 K/70 MPa in air and steam atmospheres. In all specimens, the creep rate gradually decreases after loading and then almost keeps a constant rate in transient creep stage, followed by creep acceleration. These creep curves in steam have a close overlap with the curves in air. The base steel with ρ = 43 % exhibits a minimum creep rate of 1.6 x 10^{-3} /h ,which is much higher than one (7.0 x10^{-4} /h) of the pre-aged B-doped steel with ρ = 80 %. The B-doped steel with ρ = 80 % shows high t_r of 825 h, which is more than three times higher than t_r of the base steel with ρ = 43 % (255 h). This clearly indicates that increasing ρ to 80% can substantially increase the creep resistance, resulting in the extension of creep rupture time. Both steels exhibit large ε_r of about 0.6, which demonstrates that covering grain boundaries by Laves phase dose not reduce the creep ductility. All data on creep properties of the present steel (including base and B-doped steels pre-aged at 1073 K/1200 h) at 1073 K/70 MPa are summarized in Table 1.

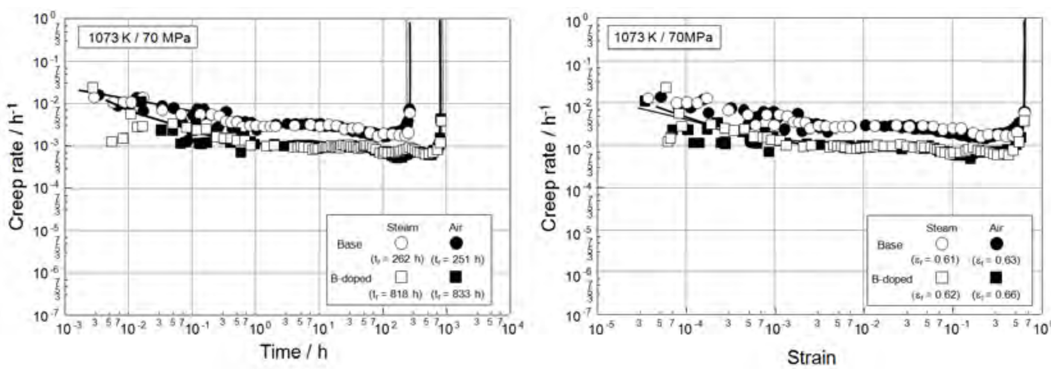

Figure 4: (a) creep rate/time curves and (b) creep rate/strain curves of base and B-doped steels (pre-aged at 1073K/240 h) at 1073K/70 MPa.

Table 1: Creep rupture time and strain of base and B-doped steels tested at 1073 K /70 MPa.

Base steel: Fe-20Cr-30Ni-2Nb (at. %)						
Heat treatment	Solution Treated		Pre-aged at 1073 K/240 h (ρ = 43%)		Pre-aged at 1073 K/1200 h (ρ = 52 %)	
Atmosphere	Steam	Air	Steam	Air	Steam	Air
Rupture time, t_r (h)	233	248	262	251	-	239
Rupture strain, ε_r (-)	0.49	0.56	0.61	0.63	-	0.56
B-doped steel: Fe-20Cr-30Ni-2Nb-0.03B (at. %)						
Heat treatment	Solution Treated		Pre-aged at 1073 K/240 h (ρ = 80%)		Pre-aged at 1073 K/1200 h (ρ = 89 %)	
Atmosphere	Steam	Air	Steam	Air	Steam	Air
Rupture time, t_r (h)	376	293	818	833	-	681
Rupture strain, ε_r (-)	0.25	0.31	0.62	0.66	-	0.59

Observation of Creep Ruptured Specimens

Figure 5 shows (a, b) stereographs of creep ruptured specimens of base steel (pre-aged at 1073 K/240 h) with ρ = 43 % in (a) steam and (b) air, together with (c) their corresponding macroscopic strain and (d, e) optical micrographs of the gauge portion in these specimens. The ruptured specimen under steam exhibits dark green color, and locally red rust surface (Fig. 5 (a)), whereas a whole surface of the specimens in air is colored gray (Fig. 5 (b)). The specimens in

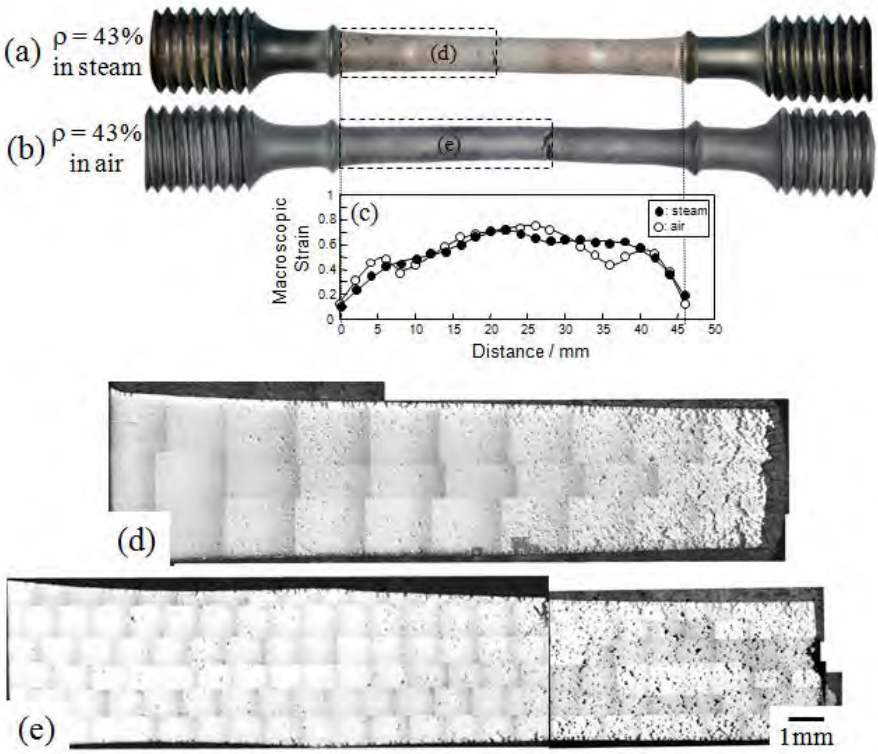

Figure 5: (a, b) Appearances of creep ruptured specimens of base steel with ρ =43% (pre-aged at 1073 K/240 h) , (c) corresponding macroscopic strain in their gauge portion and (d, e) optical micrographs of gauge portion of these specimens: (a, d) tested in steam, (b, e) tested under air.

steam exhibits a slightly smaller creep elongation than that under air. Their macroscopic strains become higher by approaching the rupture surface and reaches to 0.75 in maximum (Fig. 5 (c)). The optical micrographs indicates numerous inner cavities and their associated cracks along grain boundaries in the creep ruptured specimens. It is noteworthy that the ruptured specimens in steam shows a higher number of cavities than one under air. It is also obvious that the number of voids and cracks becomes higher by approaching the rupture surface, which indicates higher cavity density at higher creep strain.

Figure 6 show BEIs showing (a,b) the surface regions and (c,d) the inner microstructures in creep ruptured specimens of base steel with ρ = 43 %. A number of cavities and their associated cracks are observed along grain boundaries perpendicular to stress axis. The cavities are often surrounded by intergranular oxides underneath the specimen surface. The intergranular oxides with a width of several tens micrometers are present and grow over 200 μm in depth (Fig. 6 (a,b)). Its depth is lager under steam atmosphere than one under air. On the surface of γ-Fe grains, the oxide scale with a mean thickness of 10-20 μm is formed, whereas the delamination of oxide scale locally occurs by creep deformation. Inner microstructure of the crept specimen (Fig. 6 (c,d)) shows slight lower ρ, comparing with microstructures of the steel before creep test (Fig. 2 (a)), there is scarcely a change in microstructure after the creep test under air and steam atmospheres.

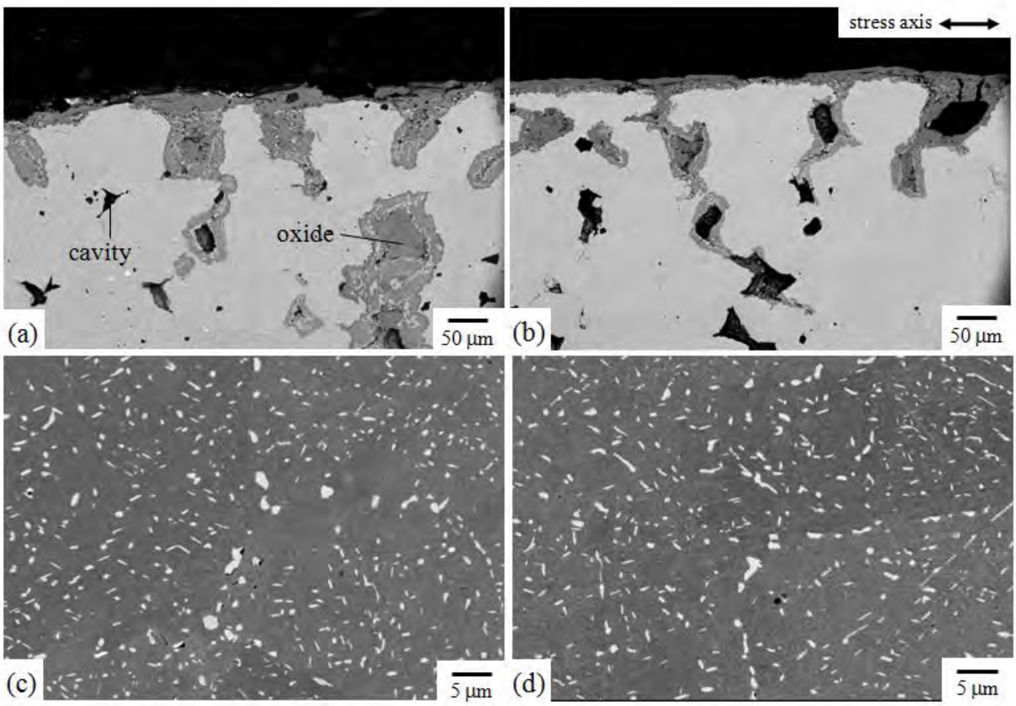

Figure 6: BEIs showing (a, b) the surface of creep ruptured specimens and (c, d) microstructure of the base steel with ρ = 43% (pre-aged at 1073 K/240 h) in (a, c) steam and (b, d) air atmospheres. Macroscopic strains of these specimens under steam and air atmospheres are 0.60 and 0.70 , respectively.

Figure 7 shows (a, b) stereographs of creep ruptured specimens of B-doped steel (pre-aged at 1073 K/240 h) with ρ = 80 % in (a) steam and (b) air atmospheres, together with (c) the corresponding macroscopic strain and (d, e) optical micrographs of the gauge portion in these specimens. The ruptured specimen of B-doped steel tested in steam exhibits dark green colored surface with local rusty colored areas (Fig. 7 (a)), as well as the base steel. In the B-doped steel, the macroscopic strain becomes higher by approaching the rupture surface. These ruptured specimen shows a localized necking (Fig. 7 (c)), comparing with base steel (Fig. 5 (a-c)) . The optical micrographs (Fig. 7 (d, e)) indicates that the ruptured specimens of B-doped steel with ρ = 80 % include the inner cavities and their related cracks, whereas their densities are much lower than those in base steel with ρ = 43 %. It is likely that the number of cavities in steam is almost same to that in air.

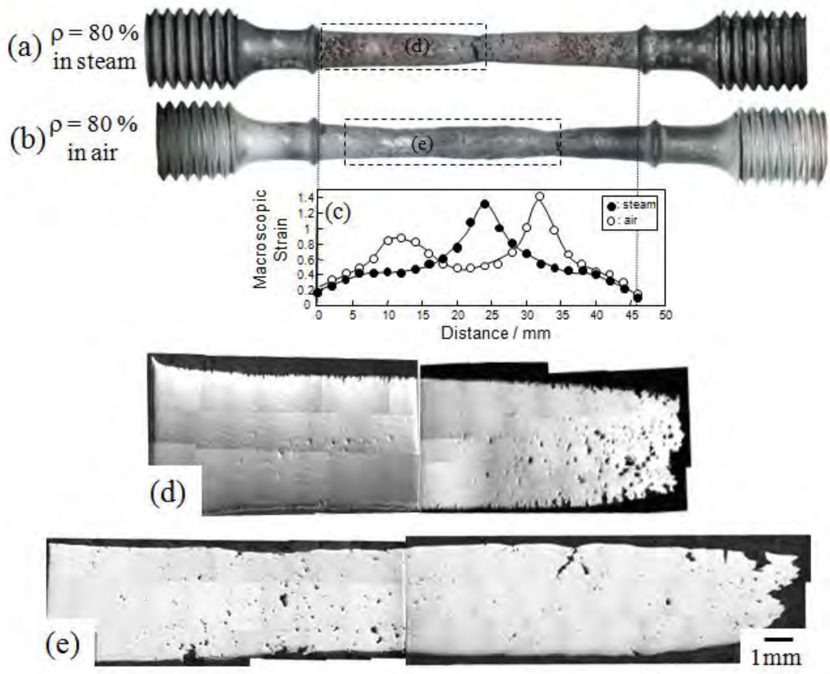

Figure 7: (a, b) Appearances of creep ruptured specimens of B-doped steel with ρ =80% (pre-aged at 1073K/240 h) , (c) corresponding macroscopic strain in their gauge portion and (d, e) optical micrographs of gauge portion of these specimens: (a, d) tested in steam, (b, e) tested in air.

Figure 8 show BEIs showing (a-d) the surface regions and (e, f) the inner microstructures in creep ruptured specimens of the B-doped steel with ρ = 80 %. The cavities and their associated cracks are scarcely observed in the specimens, even at highly strained regions. The intergranular oxides are present on the specimen surface, whereas their depth is about 100 μm on even the highly strained surface (Fig. 8(a, b)), which is much smaller than that in base steel with ρ = 43% (Fig. 6(a, b)). The depth of intergranular oxide in steam is slightly larger than that in air. Note that the oxide scale with a mean thickness of 10-20 μm is also formed on γ-Fe grains in B-doped steel, as well as base steel. There is scarcely a change in microstructure after the creep test in air and steam (Fig. 6(c, d)), comparing with microstructures of both steels before creep test (Fig. 2(c)). B-doped steel exhibits high ρ above 70% even after the creep test.

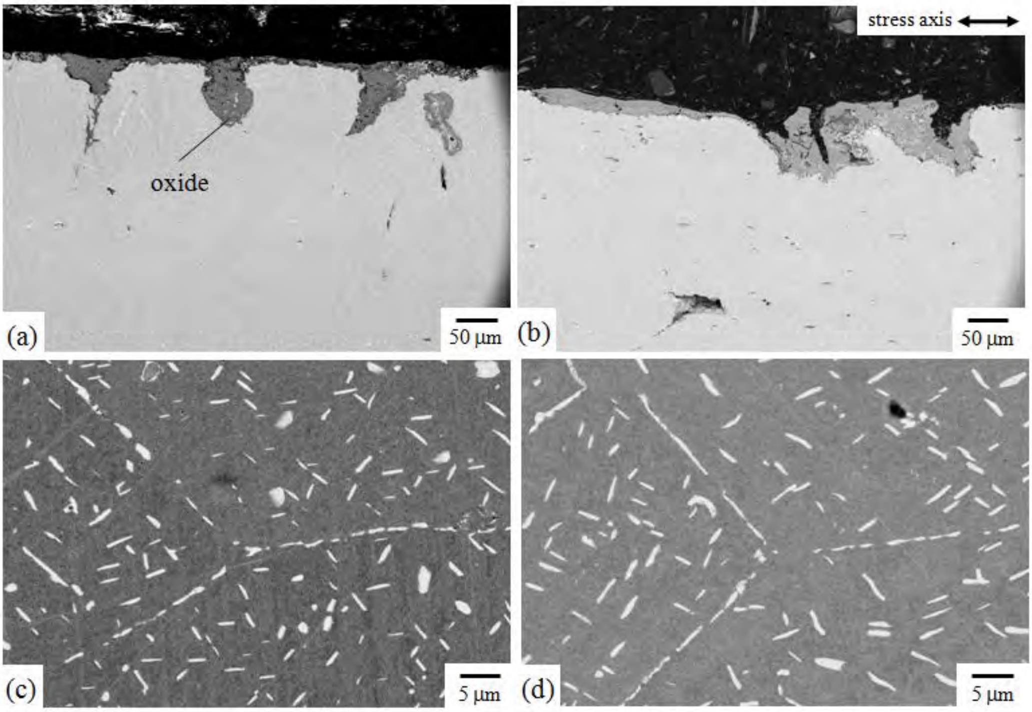

Figure 8: BEIs showing (a, b) the surface of creep ruptured specimens and (c, d) microstructure of B-doped steel with ρ = 80 % (pre-aged at 1073 K/240 h) in (a, c) steam and (b, d) air atmospheres. Macroscopic strains of these specimens in steam and air are 0.49 and 0.70 , respectively.

Figure 9 shows changes in (a, c) the depth of intergranular oxide and (b, d) the width of intergranular oxide on the specimen surface as a function of macroscopic strain (ε) in creep ruptured specimens of (a) base steel and (b) B-doped steel under both atmospheres. In the base steel with ρ = 43% in air, the depth of intergranular oxide is almost constant of about 100 μm at ε below 0.6 and increases to about 300 μm at higher ε than 0.6. In steam, the depth of intergranular oxide is almost constant at lower ε than 0.4, whereas it significantly increases at higher ε and reaches to more than 400 μm (Fig. 9 (a)). The same trend can be recognized in the width of intergranular oxide on the specimen surface (Fig. 9 (b)). In the B-doped steel with ρ = 80%, in air, the depth of intergranular oxide is almost constant of about 100 μm independent of ε, whereas it increases at higher ε than 0.4 in steam atmosphere. The depth is limited to about 200 μm in maximum, which is much smaller than one of base steel with ρ = 43 %. The width of intergranular oxide is also constant at lower ε than 0.4 and it increases to more than 200 μm at higher ε. Both depth and width in steam atmosphere is larger than that in air, which is independent of ε. These results demonstrate that steam atmosphere enhances the growth of intergranular oxides on the specimen surface at higher creep strain above 0.4, which can be suppressed by increasing ρ.

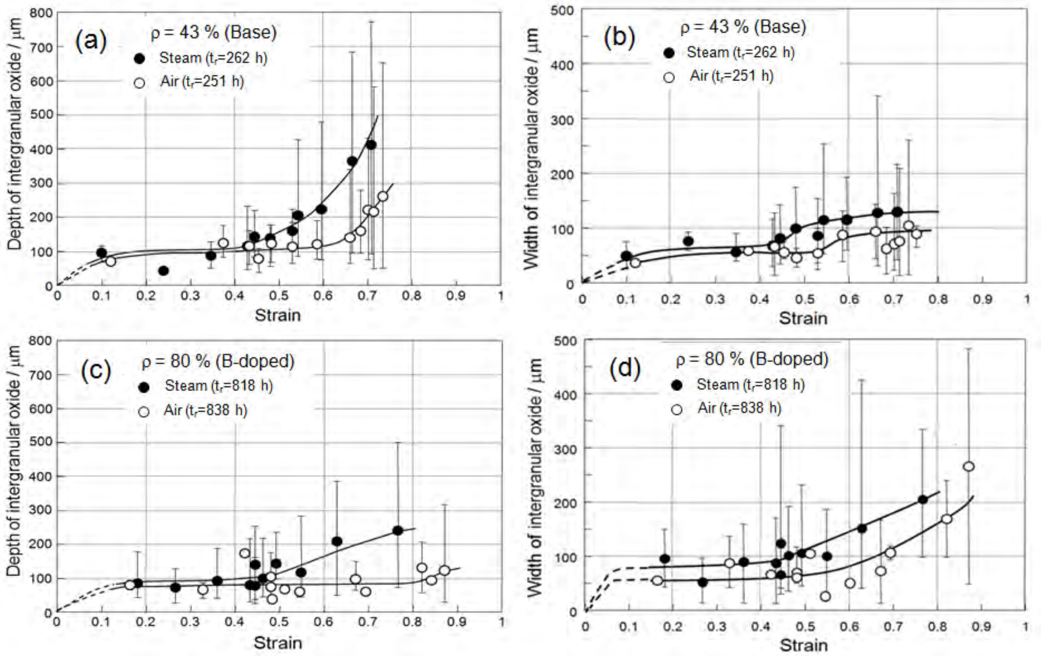

Figure 9: Change in (a,c) a depth of the intergranular oxide and (b,d) a width of the intergranular oxide on the surface as a function of macroscopic strain in creep rupture specimens of (a,b) base steel with ρ = 43 % and (c, d) B-doped steel with ρ = 80 %.

DISCUSSION

Effect of Grain Boundary Laves Phase on Creep Properties

The present creep test of the pre-aged steels with different ρ demonstrates that the pronounced extension of creep rupture time (t_r) by increasing ρ in steam atmosphere as well as air (Table 1). One of the important findings in this study is that an increasing ρ from 43 % to 80 % can reduce by one half the creep rate in the transient stage (Fig. 3, Fig. 4), resulting in a significant increase in the creep rupture life. This importantly indicates that GBPS mechanism [10-11] by stable Laves phase could work at 1073 K even in steam atmosphere. And it is also noteworthy that the pre-aged steels exhibit large creep rupture strain (ε_r) of about 0.6 under both atmospheres, which is independent of ρ. This clearly indicates that Laves phase on grain boundaries could not reduce the creep ductility. In the present study, all creep rupture times are limited below 1000 h. Thus, further creep tests beyond 1000 h should be needed to identify the effect of GBPS mechanism by Laves phase on long-term creep strength above 973 K. They are under testing at lower stress level of 50 MPa.

Effect of Steam Atmosphere on Creep Properties

Another important finding in this study is that steam atmosphere gives a slight effect on the creep rupture life and the minimum creep rate, but a few percent reduction in the creep ductility (Table 1). This ductility loss would be related to the intergranular oxidation on the specimen surface of the present steel during creep (Fig. 6 (a,b), Fig. 8 (a,b)), in particular highly strained surface (ε > 0.4). Considering that the creep acceleration stage starts at ε of about 0.4 (Fig. 3 (b), Fig. 4 (b)), the intergranular oxidation into the inside of the specimens would be enhanced by steam, in particular in the creep acceleration stage. They presumably enhance the crack propagation along grain boundaries at a late stage of creep, resulting in the ductility loss of the steel by steam.

However, the intergranular oxidation accelerated by steam atmosphere can be suppressed by sufficiently covering grain boundaries by stable Laves phase (increasing ρ above 80 %) (Fig. 9). This represents that Laves phase on grain boundaries would reduce the environment effect on creep properties of the present steel. Such inhibiting the intergranular oxidation by Laves phase on grain boundaries would be associated with the formation of voids and cracks (connecting each other) along grain boundaries during creep. Optical micrographs of the ruptured specimens (Fig. 5 (d,e), Fig. 7 (d,e)) clearly indicate that increasing ρ can inhibit the formation of voids and their associated cracks. These results can be understood on the basis that in creep acceleration stage, localized deformation around grain boundaries without precipitates of Laves phase might accelerate the formation of voids, which connects each other and makes it cracks propagating along grain boundaries to the surface of specimens, thereby extensively enhancing the intergranular oxidation. To confirm this speculation, further quantitative analyses for the local deformation and the void formation along grain boundaries should be needed. They must await future research to understand a role of Laves phase on grain boundaries in creep deformation under steam atmosphere.

Based on above results, it can be summarized that the stable Laves phase sufficiently covering grain boundaries can increase the creep long-term creep rupture strength and suppress the intergranular oxidation during creep even in steam atmosphere. Therefore, GBPS mechanism is a key strengthening mechanism for use of innovative advanced ultra-super critical (A-USC) thermal power plant to be operated above 973 K.

CONCLUSIONS

Creep of a novel austenitic heat resistant steel of Fe-20Cr-30Ni-2Nb (at.%) strengthened by grain boundary Laves phase at 1073 K under steam and air atmospheres (t_r < 1000 h) has been examined. The conclusions are as follow:

(1) Sufficiently covering grain boundaries by stable Laves phase (ρ > 80 %) can increase the creep resistance without a loss of creep ductility at 1073 K, resulting in the significant extension of creep rupture life in steam as well as air. The creep rupture strain slightly decrease by steam atmosphere.

(2) The intergranular oxidation and its related crack propagation along grain boundaries would be enhanced by steam atmosphere at higher creep strain than 0.4 in the creep acceleration stage. Its effect can be suppress by increasing ρ.

(3) *Grain Boundary Precipitation Strengthening* is a key mechanism to not only improve the creep long-term creep rupture strength and but reduce the environment effect on creep properties in steam atmosphere.

ACKNOWLEDGMENTS

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