ZIG-ZAG GRAIN BOUNDARIES AND STRENGTH OF HEAT RESISTING ALLOYS

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ABSTRACT

The role of zig-zag grain boundaries with coarse carbides for the deformation and fracture behaviors in creep was studied in an austenitic heat resisting steel having wide range of hardness. With increase in hardness, the rupture strength increases until maximum strength is reached at some critical value of hardness and then decreases in spite of decrease in creep rate. From the analysis of fracture mode, it was clarified that the transition of fracture mode from ductile to brittle with increasing hardness is responsible for this variation in the rupture strength. Zig-zag grain boundaries with coarse carbides can remarkably increase the rupture strength by increasing the critical hardness as a result of preventing the formation and the growth of cracks and cavities along grain boundaries.

It was found that the combination of zig-zag grain boundaries and high hardness is quite beneficial to improve the rupture strength and that for this purpose, two step cooling process, i.e. direct furnace cooling to about 900°C after solution heating followed by rapid cooling, is very effective for both Fe base and Ni base superalloys. For Ni base superalloy containing low carbon, however , directly aging treatment to about 900°C is more useful than two step cooling process.

Introduction

In order to improve the creep rupture strength it is important to prevent the nucleation and growth of cracks and cavities occurred in grain boundaries because the creep fracture at elevated temperatures is apt to occur by grain boundary cracking. Recent several works on high carbon austenitic heat resisting steels(1)∿(4) have shown that zig-zag grain boundaries with coarse carbides are quite effective to prevent the grain boundary cracking, i.e. to strengthen the grain boundary. It has been also suggested that the efficiency of zig-zag boundaries must be examined with close relation to the strength of grains (hardness), and that an excellent rupture strength could be obtained if both grain and grain boundary are strengthened in keeping a good balance. Few studies, however , have been made in this point of view.

In this study, the role of zig-zag grain boundaries for the creep rupture strength was investigated on some Fe base and Ni base heat resisting alloys having various levels of hardness. And some heat treatments were found which are useful to improve the creep rupture strength by virtue of strengthening of both grain and grain boundary.

Experimental Procedures

An austenitic heat resisting steel containing high carbon and phosphorus, 20-11P, and Inconel 751 were selected for this study and their chemical compositions are listed in Table 1. Extremely large age-hardening can be obtained by homogeneous precipitation of $M_{23}C_6$ type carbides in 20-11P and of gamma prime particles in Inconel 751, whereas $M_{23}C_6$ type carbides precipitate along grain boundaries in both alloys (4)(5).

Three cooling rates after solution heating, water quenching(WQ), air cooling (AC) and furnace cooling(FC), were adopted to change the size of grain boundary carbides and the configuration of grain boundary. Average cooling rate in the range of 1100°C ∿ 700°C during air and furnace cooling from solution temperature of 1200°C is about 20°C/sec and 10°C/min, respectively.

Creep rupture tests were carried out at 700°C in 20-11P and at 750°C in Inconel 751. The size of smooth bar specimen is 5.5ϕ×30 mm G.L. and the root diameter, root radius and notch depth of V-notched one are 5.5ϕ, 0.5 mm and 0.92 mm respectively.

Experimental Results and Discussion

Creep Rupture Strength and Cooling Rate after Solution Heating

Microstructures of both alloys are shown in Figure 1, which were cooled at various rates after solution heating at 1200°C and then aged at 750°C. Grain boundaries in furnace cooled specimens (Figure 1-c,e) are irregularly serrated by coarse carbides precipitated along them during furnace cooling, whereas they are straight in water quenched ones (Figure 1-a) and slightly serrated in air cooled ones (Figure 1-b). The morphology of dendritic coarse carbides shown in Figure 1-d suggests that rod-like carbides grow with a certain orientation to matrix on either side of boundary. The grain boundaries of furnace -cooled Inconel 751 are less serrated owing to smaller boundary carbides as compared with 20-11P.

Variations in creep rupture strength and aged hardness with cooling rate are listed in Table 2. In both alloys, the rupture strength of furnace cooled specimen is much higher than that of water quenched one in spite of lowest hardness due to coarse precipitates within grains nucleated at higher temperature range. The slightly higher rupture strength than that of furnace cooled specimen can

Table 1. Chemical compositions of alloys (wt%)

alloy	C	S	P	Ni	Cr	Mo	Al	Ti	Nb+Ta	Fe	B
20 - 11 P	0.31	0.016	0.20	10.51	19.55	1.98	—	—	—	bal.	0.005
Inconel 751	0.06	0.007	—	bal.	14.53	—	1.07	2.39	0.96	6.52	—

Table 2. Variation in rupture strength and aged hardness with cooling rate.

alloy	heat treatment	1000hr rupture strength	test temperature	hardness before test (20kg)
20 - 11 P	WQ + 750°C×6hr	13.5 kg/mm^2	700°C	338
	AC + "	23.5 "	"	358
	FC + "	22.0 "	"	283
Inconel 751	WQ + 750°C×24hr	13.3 "	750°C	366
	AC + "	19.1 "	"	358
	FC + "	23.8 "	"	314

be obtained in 20-11P by air cooling as a result of slightly serrated boundaries and the highest hardness.

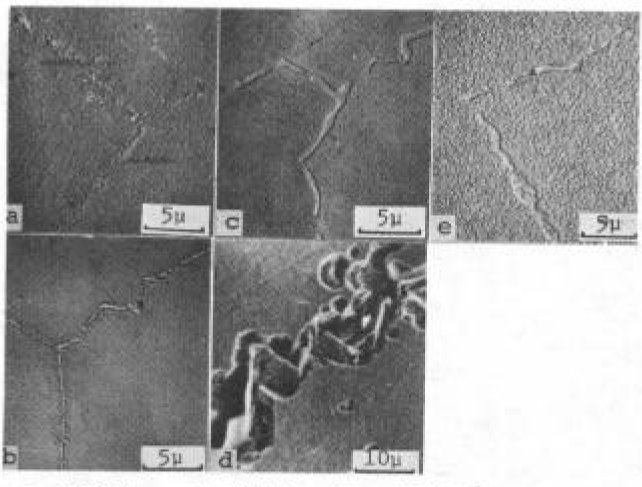
In water quenched specimens wedge-type cracks leading to entire rupture are predominant as shown in Figure 2-a, whereas in furnace cooled ones (Figure 2-b) rupture takes place by the growth and coalescence of many cavities and no wedge-type cracks appear. Moreover, cavities are isolated each other and the coalescence of them are prevented by zig-zag boundaries.

The shear stress σ_s required to initiate a wedge-type crack at triple junctions of grain boundaries is inversely to the square root of sliding boundary length L as given in equation (1) by McLean (6).

$$\sigma_s \geqq \sqrt{12\gamma G / \pi L} \quad \cdots\cdots\cdots \quad (1)$$

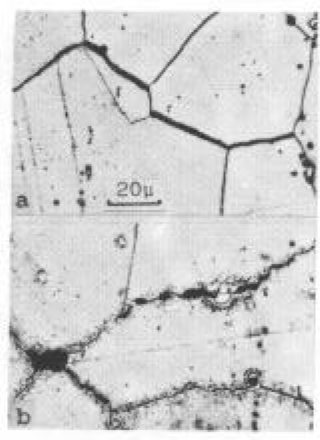
where γ is the new surface energy of crack and G is shear modulus. When grain boundaries are irreguraly serrated, the length of sliding boundary L can be reduced from the distance between two triple junctions to the length of one side of zig-zag and consequently the shear stress σ_s is so remarkably increased that the initiation of wedge-type cracks can be sufficiently prevented. In addition to this, zig-zag boundaries with coarse carbides can also suppress the growth of cavities owing to the higher surface energy at fracture as expected from Figure 1-d.

The above results reveal that zig-zag boundaries with coarse carbides are effective to imprave the creep rupture strength by preventing the formation and rapid propagation of wedge-type cracks as well as the growth of cavities on boundaries, i.e. by strengthening grain boundaries.



a 20-11P 1200°C×1hr WQ + 750°C×6hr AC
b " " AC + "
cd " " FC + "
e Inconel 751 1200°C×2hr FC + 750°C×24hr AC

Figure 1. Microstructures of alloys cooled by various rates after solution heating.



a water quenching
b furnace cooling

Figure 2. Cracks along grain boundaries in 20-11P (700°C-30 kg/mm^2. Stress axis is vertical.)

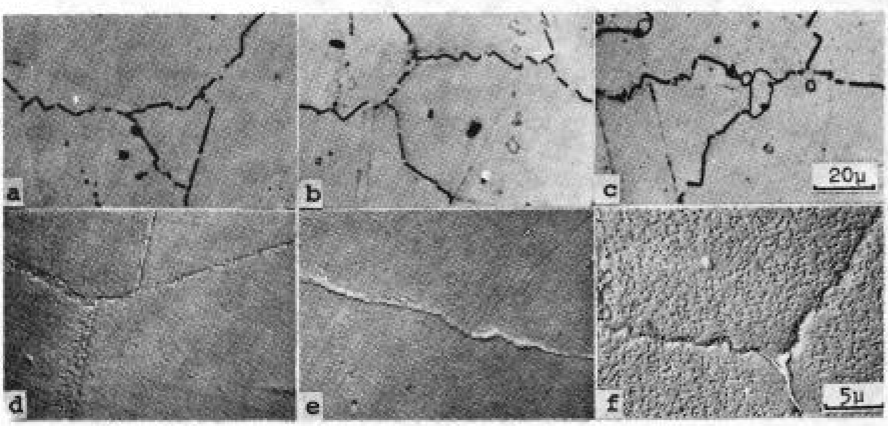
Rupture Strength and Multiple Heat Treatment

Furnace cooling to room temperature after solution heating strengthens grain boundaries but it results in the remarkable decrease in hardness due to coarse precipitates within grains nucleated during cooling. Rapid cooling is necessary to strengthen grains themselves by finely dispersed precipitates. Then, two step cooling process was applied to both alloys, i.e. direct furnace cooling to various temperatures from solution temperature followed by rapid cooling, WQ or AC. Figure 3 shows the configuration of grain boundaries of two step cooled and aged specimens. As the starting temperature of rapid cooling decreases, grain boundary carbides grow more coarsely and the formation of zig-zag boundaries is almost completed at about 900°C with little decrease of hardness. And consequently rupture life shows a peak value at about 800°C in 20-11P and at about 950°C in Inconel 751 as shown in Figure 4. Further decrease of starting temperature, however , results in the lower rupture strength as a result of the marked decrease in hardness.

It was established from above results that a noticeable improvement in rupture strength can be attained by virtue of a good combination of higher strength in both grain and grain boundary by two step cooling process in which rapid cooling is started at 900 ∿ 800°C in 20-11P and at 950°C in Inconel 751.

In Inconel 751 containing low carbon, however , grain boundary carbides are smaller and thus grain boundaries are less irregularly serrated as compared with 20-11P. In order to obtain more irregularly serrated boundaries with coarser carbides, directly aging treatment is employed for Inconel 751. The holding time at directly aging temperature was established for 6hr because longer holding time causes the coalescence of carbides and grain boundaries without carbides appear . The maximum rupture strength can be obtained after directly aging at 900°C in spite of the large decrease in hardness as shown in Figure 5. Grain boundaries of directly aged specimen are serrated to a great extent than two step cooled specimens as shown in Figure 6-a. Figure 6-b also shows the microstructure of triple heat treatment (THT) recommended for high rupture strength in this alloy of which grain boundaries are occupied by large blocky carbides but not serrated after intermediate aging at 850°C for 24hr.

The process by which zig-zag grain boundaries are formed by coarse carbides can be considered as follows; During furnace cooling or directly aging at high temperature range, small number of carbides nucleate on grain boundaries and grow rapidly toward matrix with a certain orientation to it. And consequently



a	20-11P	1200°C×1hr→FC→1000°C→AC	+ 750°C×6hrAC
b	"	" 900°C "	+ "
c	"	" 800°C "	+ "
d	Inconel 751	1200°C×2hr→FC→1000°C→WQ	+ 750°C×24hrAC
e	"	" 950°C "	+ "
f	"	" 900°C "	+ "

Figure 3. The configuration of grain boundary of two step cooled specimens.

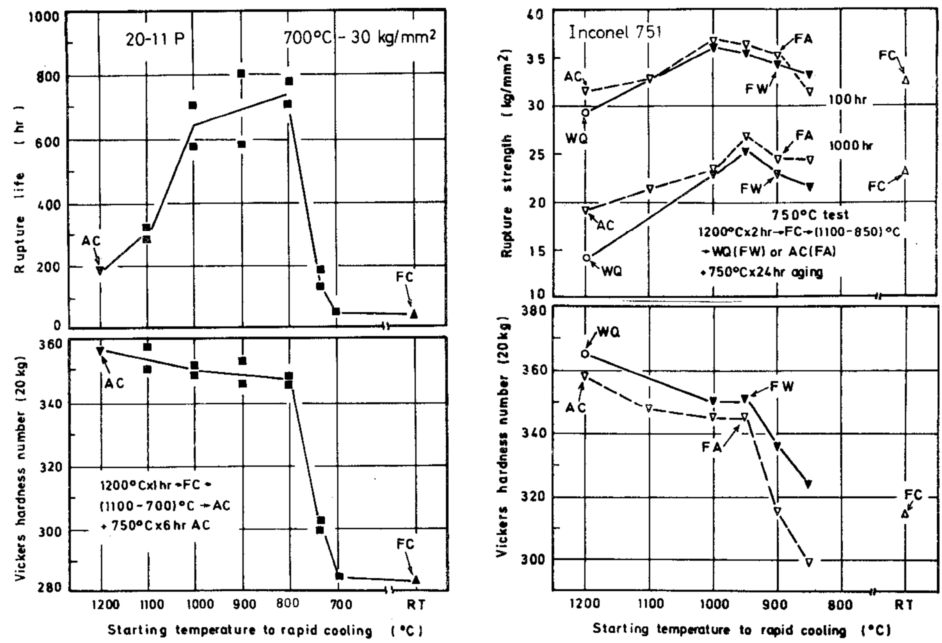


Figure 4. Rupture strength and aged hardness of two step cooled specimens.

grain boundary migration occurs so as to minimize the total length of grain boundaries and zig-zag boundaries can be formed.

On the other hand, when specimens are re-heated after rapid cooling to room temperature as in the case of THT, grain boundaries remain as a straight line because a great number of carbides nucleate at lower temperature range during re-heating and they only grow to large blocky particles during intermediate aging.

The maximum rupture strengths of Inconel 751 obtained by various multiple heat treatments are listed in Table 3. This suggests that it has marked effect on the improvement of rupture strength for grain boundaries to be not only occupied by coarse carbides but also to be serrated irregularly.

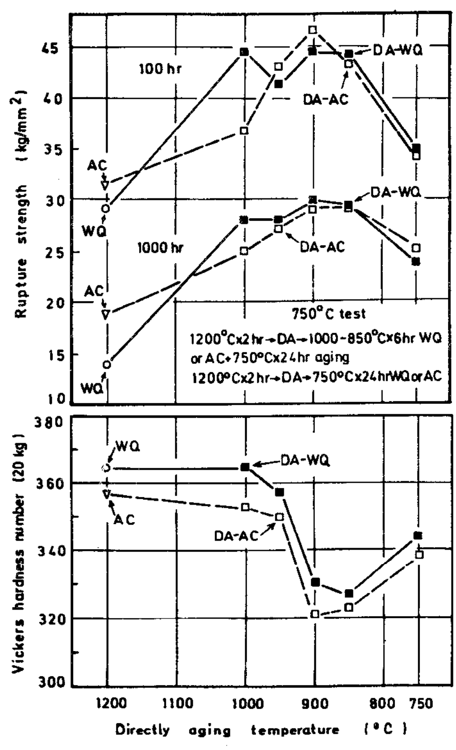
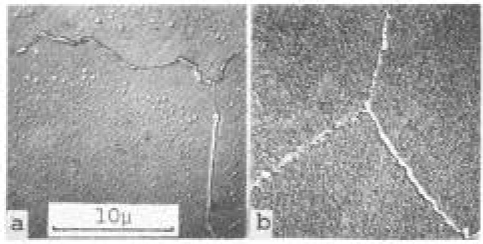


Figure 5. Variation in rupture strength and aged hardness of directly aged Inconel 751.

Table 3. 1000hr rupture strength at 750°C of Inconel 751.

heat treatment	strength
Two step cooling 1200°C×2hr+FC→950°CAC + 750°C×24hrAC	26.5 kg/mm^2
Directly aging 1200°C×2hr+DA→900°C× 6hrAC + 750°C×24hrAC	29.8 "
Triple heat treatment 1150°C×2hrAC + 850°C× 24hrAC + 700°C×20hrAC	26.5 "



a 1200°C×2hr→DA→900°C×6hrAC+750°C×24hrAC
b 1150°C×2hrAC+850°C×24hrAC+700°C×20hrAC

Figure 6. Microstructures of Inconel 751.

Creep Rupture Properties and Strength of Grain and Grain Boundary

Further study on creep rupture properties was made using 20-11P heat-treated by various processes. Three processes, i.e. water quenching WQ, air cooling AC and two step cooling FA (furnace cooling to 900°C and subsequent air cooling) followed by aging at 600 ∿ 900°C, were employed. Two solution heating conditions, 1200°C×1hr and 1150°C×1hr, were also employed to change the size of grains in these processes. Average diameter of grains was about 200 μm and 60 μm respectively.

The rupture lives and elongations of smooth specimens tested at 700°C under 30 kg/mm^2 are plotted against hardness before tests in Figure 7. The arrows in figures show the change of hardness during tests. As hardness raises, the rupture life increases up to some critical hardness Hv* corresponding to each process, but suddenly decreases in the range above Hv*. The critical hardness raises with strengthening of grain boundary and thus the highest rupture life can be obtained by two step cooling process. Consequently, it was found that zig-zag boundaries are more effective on improving rupture strength in the hardness range above Hv*-WQ rather than below it. The critical hardness Hv*-WQ and the maximum life of water quenched specimens also increase with decreasing grain size. This shows that fine grains have an equivalent effect to zig-zag boundaries so far as the increasing in rupture strength.

Rupture elongation decreases with hardness and it is notable that rupture ductility is extremely poor in the hardness range above each critical value regardless of cooling process.

Minimum creep rate under constant stress shown in Figure 8 is inversely changed to hardness over whole range of hardness tested. In higher hardness level above about Hv250, minimum creep rates of two step cooled specimens are always lower than those of water quenched ones. This suggests that zig-zag boundaries can sufficiently inhibit grain boundary sliding.

The relationship between rupture lives and minimum creep rates summarized in Figure 9 falls in two groups. One is an ordinary relationship along A-B-C-D and A'-B'-D' (referred to type I) where rupture lives are proportional to a reciprocal of minimum creep rates. Another is an unusual relationship along B-E, C-F, D-G and

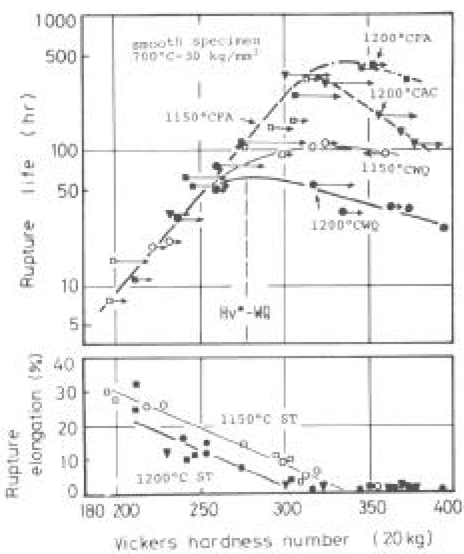


Figure 7. Variation in rupture life and elongation of smooth 20-11P specimens with hardness. (700°C, 30 kg/mm^2)

B'-E' (referred to type II) where rupture lives decrease in spite of decreasing of minimum creep rates. The rupture lives are essentially a linear function of minimum creep rates in type I whereas they strongly depend on grain boundary strength in type II . From these results it can be deduced that the determining process of rupture life is quite different for each type.

Figure 10 shows the fractographs of fracture surfaces and Figure 11 shows the average wedge length and density of wedge-type cracks remained in raptured smooth specimens. The measurement of crack dimensions was carried out at longitudinal cross section within 3 mm from fracture surface. In water quenched specimens in which only wedge-type cracks were found over whole range of hardness as shown in Figure 10-a ∿ c, many small cracks are formed in lower hardness region showing that grain boundary fracture may progress in a ductile manner in this region. As hardness raises beyond about Hv300, the density and length of wedge-type cracks again decrease to extremely low values as shown in Figure 11 showing that fairly brittle fracture occurs by rapid propagation of a few cracks. The ductile-brittle transition of fracture mode with increase in hardness corresponds to the transition of rupture life dependency on minimum creep rate from type I to type II . Thus it can be deduced that in type I where ductile fracture is predominant, the rupture life may be controlled by the growth process of cracks and therefore it can increase with decreasing of creep rate, i.e. strengthening of grain itself. On the contrary, in type II where fairly brittle fracture occurs and the nucleation of cracks may be a controlling process of rupture life, the increase of stress concentration at triple junctions with increase in hardness facilitates crack nucleation and consequently causes the premature fracture.

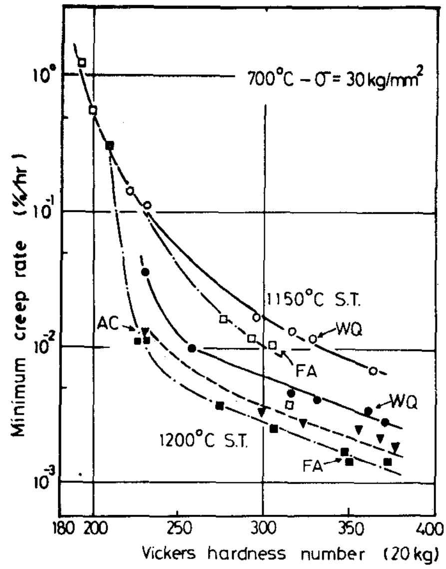


Figure 8. Variation in minimum creep rate as a function of hardness in 20-11P. (700°C - 30 kg/mm^2)

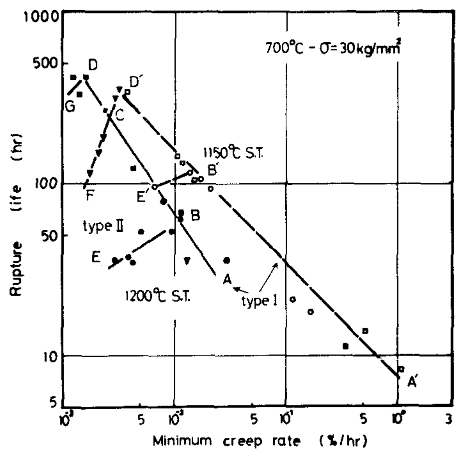
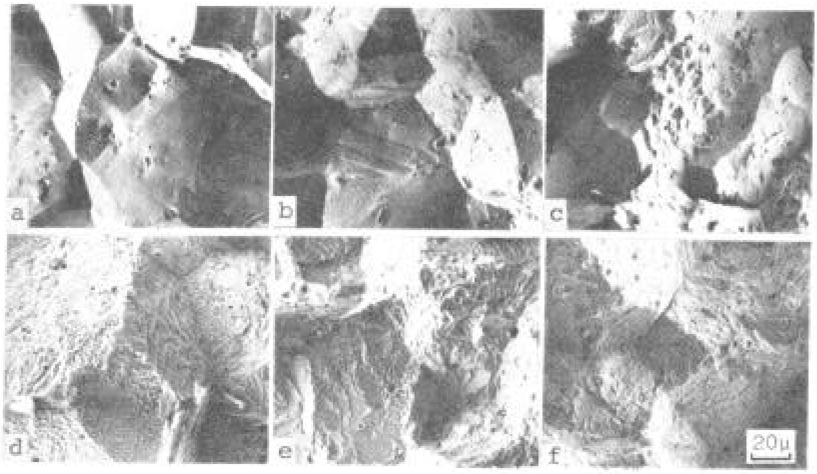


Figure 9. Relationship between rupture life and minimum creep rate in 20-11P. (700°C - 30 kg/mm^2)

On the other hand, fracture of two step cooled specimens takes place mainly by the growth and coalescence of cavities as shown in Figure 10 d ∿ f. Zig-zag boundaries with coarse carbides can shift the ductile-brittle transition to higher hardness as a result of preventing the formation of wedge-type cracks and thus improve the rupture strength remarkably. Since fine grain has a similar effect to strong grain boundary, a critical hardness is increased to higher value and higher rupture strength can be obtained in fine grained specimens.

The rupture lives of notched specimens vary with hardness in a similar manner to those of smooth ones, but the critical hardness at which the maximum



a	20-11P	1200°C 1hr	WQ,	Hv393
b	"	"	" ,	Hv361
c	"	"	" ,	Hv316
d	"	"	FA,	Hv370
e	"	"	" ,	Hv345
f	"	"	" ,	Hv305

Figure 10. Fractographs of ruptured smooth specimens. (700°C, 30 kg/mm^2)

life can be obtained is lowered by notch as shown in Figure 12. From the analysis of fracture mode it was clarified that notched specimens fracture in a brittle mode even at lower hardness region where smooth one fractures in a ductile mode and that the ductile-brittle transition also occurs at lower hardness. This shift of the critical hardness leads to notch strengthening at lower hardness, while it leads to notch weakening at higher hardness. Figure 13 illustrates a variation of notch rupture life ratio as a function of hardness. In higher hardness region where brittle fracture may occur, stress concentration by notch results in notch weakening owing to a faster rate of crack nucleation, whereas in lower hardness region plastic constraint by notch causes notch strengthening by lowering the growth rate of cracks. All results mentioned above emphasize that in order to improve the creep rupture strength the grains must be strengthen in keeping a good balance with the strength of grain boundary and it

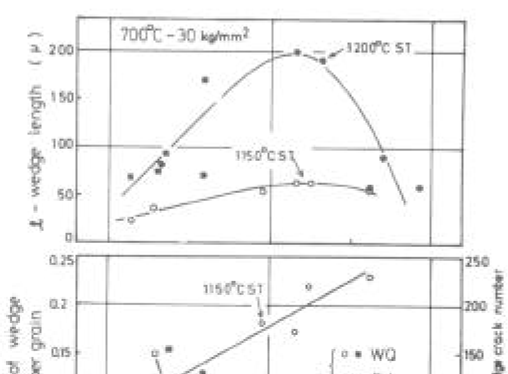


Figure 11. Wedge length and density of wedge-type cracks as a function of hardness in 20-11P smooth specimens. (700°C, 30 kg/mm^2)

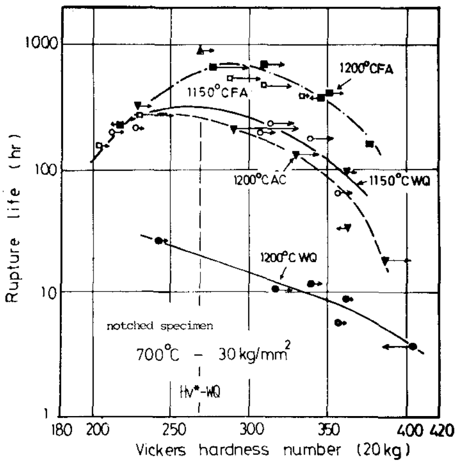


Figure 12. Variation in rupture life of notched 20-11P specimen with hardness. (700°C, 30 kg/mm^2)

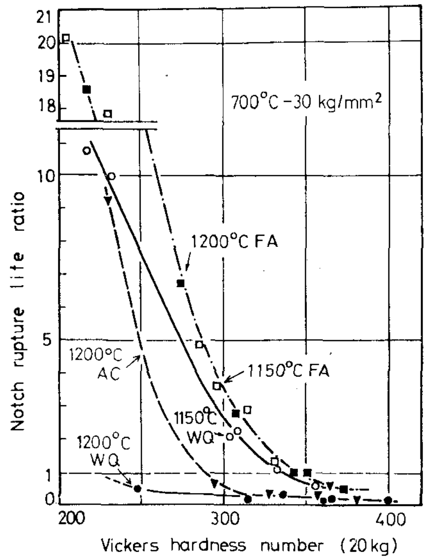


Figure 13. Notch rupture life ratio as a function of hardness in 20-11P. (700°C, 30 kg/mm^2)

has a rather harmful effect to strengthen only the grains.

Conclusion

The effect of zig-zag grain boundaries with coarse carbides on the deformation and fracture mode in creep was investigated. Effective heat treatments for improving of rupture strength were also found in some Fe base and Ni base heat resisting alloys.

1) The zig-zag boundaries with coarse carbides have a marked effect on the prevention of the formation and rapid propagation of wedge-type cracks as well as the coalescence of cavities on grain boundaries; in other words, they have a good resistance to grain boundary cracking and contribute to the strengthening of grain boundaries.
   The finely dispersed precipitates within grains increase hardness and reduce the creep rate, i.e. they contribute to the strengthening of grains themselves.

2) With increase in hardness (strength of grain) the rupture strength increases due to reduced creep rate until a peak value is reached at some critical hardness Hv* but it decreases abruptly in the range above Hv* in spite of a further reducing of creep rate.
   Such a variation in rupture strength with increase in hardness has a close correlation to the transition of fracture mode from ductile one in range below Hv* to brittle one in range above Hv*.
   Serrated boundaries have a marked supressed effect on the brittle grain boundary cracking and shift a critical hardness to higher value and consequently increase the rupture strength.

3) For this reason, in order to improve the creep rupture strength, the hardness must be increased in keeping a good balance with the grain boundary strength and it has a rather harmful effect on the rupture strength to increase only the hardness beyond a critical value in the alloys having weaker strength of grain boundaries.

4) Grain boundaries can be serrated remarkably by furnace cooling or holding at higher temperature range after solution heating. Hardness is lowered, however , if furnace cooling is performed to room temperature or holding temperature is too low.

On the other hand, only straight boundaries with coarse carbides are formed after rapid cooling to room temperature followed by high temperature holding.

Thus, in order to obtain a good combination of higher strength of both grains and grain boundaries and increase the rupture strength, two step cooling, i.e. furnace cooling to about 900°C from solution temperature followed by rapid cooling, or directly aging at about 900°C are found to be much more useful processes.

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