

# Effects of high-temperature ageing on the creep-rupture properties of high-tungsten cobalt-base superalloys

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The effects of high-temperature ageing on creep-rupture properties were studied using cobalt-base superalloys containing about 14–20 wt % tungsten (W) at 1089 K (816 °C) and 1311 K (1038 °C) in air. A high-temperature ageing for 1080 ks at 1273 K after solution treatment caused grain-boundary and matrix precipitates of W solid solution and carbide phases in these alloys, and grain boundaries were serrated especially in the alloys with higher W content. The high-temperature ageing largely improved the rupture life in the alloys with higher W content, particularly under lower stresses at 1089 K, whereas it caused the creep ductility to decrease a little in the alloy containing 20% W. The high-temperature ageing also improved the rupture life without decreasing creep ductility in these alloys under higher stresses at 1311 K. Under the same ageing conditions of 1080 ks at 1273 K, the initiation of grain-boundary cracks was retarded in the solution-treated and aged specimens, as well as in the aged specimens with serrated grain boundaries, for the alloys with higher W content at both 1089 and 1311 K. A large amount of grain-boundary serration also occurred in the non-aged specimens of the alloys with higher W content during creep at 1311 K, and contributed to the strengthening of the alloys. The solution-treated and aged specimen had almost the same rupture strength as the aged specimens with serrated grain boundaries in these cobalt-base alloys. The rupture strength of the solution-treated and aged specimens largely increased with increasing W content under the lower stresses at 1089 K and under the higher stresses at 1311 K. A ductile grain-boundary fracture surface, which was composed of dimples and grain-boundary ledges associated with grain-boundary precipitates, was observed in the solution-treated and aged specimens, as well as in the aged specimens with serrated grain boundaries at both 1089 and 1311 K. The fracture surface of the non-aged specimens was a brittle grain-boundary facet at 1089 K, but it became a ductile grain-boundary fracture surface, as serrated grain boundaries were formed owing to grain-boundary precipitates occurring during creep at 1311 K.

## 1. Introduction

In a previous study on the cobalt-base L-605 alloys [1, 2] it was found that high-temperature ageing for 1080 ks at 1273 K (1000 °C) caused a large amount of grain-boundary and matrix precipitates of tungsten (W) solid solution and  $M_6C$  carbide, and led to an improved rupture life without decreasing creep ductility in high-temperature creep. Furthermore, a ductile grain-boundary fracture, similar to the one observed in the specimen with serrated grain boundaries, was found to occur also in the aged specimens with originally straight grain boundaries in this alloy [2]. Thus the strengthening mechanisms in the aged specimens were attributed to the precipitation hardening of the matrix, and also to the strengthening of grain boundaries by the precipitates which were formed under high-temperature ageing [1, 2].

In the cobalt-base superalloys containing about 14–20 wt % W (3), the rupture life in the solution-treated specimens largely increased with increasing W

content at 1311 K (1038 °C); however the rupture life at 1089 K (816 °C) was not improved by increasing the level of W, as the Laves phase which is believed to be harmful to ductility [4] precipitated during creep at 1089 K. Therefore, pre-ageing at 1273 K may cause the precipitates of tungsten solid solution and carbide phases, and may improve the creep-rupture properties also in these alloys at 1089 K.

In this study, the effects of high-temperature ageing for 1080 ks at 1273 K on creep-rupture properties were investigated using the L-605 type cobalt-base superalloys containing about 14–20 wt % tungsten (W) at temperatures of 1089 (816 °C) and 1311 K (1038 °C) in air. The precipitated phases caused by high-temperature ageing were identified by X-ray diffractometry (XRD). The initiation of grain-boundary cracks during creep was then examined in those alloys. For comparison, the specimens with serrated grain boundaries [1, 2, 5] were also aged and creep-tested under the same conditions.

TABLE I Chemical composition of the cobalt-base superalloys (wt %)

Alloy	C	Cr	Ni	W	Fe	Mn	Si	P	S	Co
14 W	0.07	19.82	9.83	14.37	2.22	1.46	0.19	<0.005	0.002	Balance
17 W	0.06	19.40	9.89	17.20	2.37	0.76	0.19	0.001	<0.001	Balance
20 W	0.06	19.05	9.54	19.74	2.28	0.77	0.19	0.003	0.001	Balance

## 2. Experimental procedure

Table I shows the chemical composition of the cobalt-base superalloys, containing about 14–20 wt % tungsten, used in this study [3]. The alloys were supplied by Mitsubishi Materials Company Ltd, (Tokyo) in the form of hot-forged bars of 20 mm diameter. The alloy bars were solution-treated to obtain creep-rupture specimens of about the same grain size with straight grain boundaries. The specimens with serrated grain boundaries were obtained by the heat treatments described in [5]. Part of the specimens were then aged for 1080 ks at 1273 K (1000 °C) to develop matrix and grain boundary precipitates of tungsten (W) solid-solution and carbide phases [2]. Table II shows the heat treatments, the matrix hardness and the grain size in specimens of the cobalt-base superalloys. The matrix hardness of the aged specimens increased with increasing W content, while there was only a small increase in matrix hardness with W content in the solution-treated specimens [3]. The solution-treated and aged specimens had almost the same matrix hardness as the aged specimens with serrated grain boundaries in these alloys, except in the aged specimens of the 14 W alloy. The alloys are hereafter referred to as “14 W alloy”, “17 W alloy” or “20 W alloy” according to tungsten content.

The heat-treated specimens were machined into creep-rupture test pieces of 5 mm diameter and 30 mm gauge length. Creep-rupture experiment were carried out using creep-rupture equipment of the single-lever type at 1089 K (816 °C) and 1311 K (1038 °C) in air. Microstructures of the heat-treated and ruptured specimens, and the fracture surfaces of the ruptured specimens, were examined with both optical and scanning electron microscopes (SEM). The precipitated phases in the heat-treated or ruptured specimens were also identified by XRD. The fractal dimension of the grain boundary was also examined for some specimens by the vertical section method [6–9] used in a previous study [10].

## 3. Results and discussion

### 3.1. Microstructures of heat-treated specimens

Fig. 1 shows optical micrographs of the heat-treated specimens in the 14 and 20 W alloys. The matrix of the as-solution-treated specimens is composed of  $\beta$ -Co (fcc) solid solution, although a small number of residual precipitates are visible in these specimens (Fig. 1a and b) [3]. The grain-boundary and matrix precipitates are formed by ageing for 1080 ks at 1273 K, and the amount of precipitates increases with increasing tungsten (W) content of the

TABLE II Heat treatments, grain size and matrix hardness in specimens of cobalt-base superalloys

Alloy	Heat treatment	Matrix hardness Hv (kg mm <sup>-2</sup> )	Grain diameter (μm)
14 W	1473 K, 7.2 ks → WQ (solution treatment)	253	255
	1473 K, 7.2 ks → WQ + 1273 K, 1080 ks → AC	326	255
	1473 K, 3.6 ks, FC → 1323 K, 72 ks → WQ + 1273 K, 1080 ks → AC (serrated grain boundary)	301	260
17 W	1503 K, 7.2 ks → WQ (solution treatment)	264	246
	1503 K, 7.2 ks → WQ + 1273 K, 1080 Ks → AC	344	246
	1503 K, 7.2 ks—FC → 1323 K, 10.8 ks → WQ + 1273 K, 1080 ks → AC (serrated grain boundary)	338	249
20 W	1573 K, 7.2 ks → WQ (solution treatment)	269	273
	1573 K, 7.2 ks → WQ + 1273 K, 1080 ks → AC	379	273
	1573 K, 7.2 ks—FC → 1323 K, 3.6 ks → WQ		
	+ 1273 K, 1080 ks → AC (serrated grain boundary)	372	280

WQ, water-quenched; AC, air-cooled; FC, furnace-cooled; Hv, Vickers hardness number.

specimens (Fig. 1c and d). Grain boundaries are serrated owing to grain-boundary precipitates, especially in the specimen with higher W content (Fig. 1d). As shown below, the grain-boundary and matrix precipitates were identified as tungsten (W) solid solution and carbide phases by XRD [3]. Similar precipitates were observed in the aged specimen of the 17 W alloy and in the aged specimens with serrated grain boundaries of these alloys.

Table III shows XRD data of the aged specimens in the cobalt-base superalloys. In addition to strong diffraction peaks of  $\beta$ -Co (fcc) solid-solution matrix, some diffraction peaks of tungsten (W) solid solution and weak diffraction peaks of M<sub>6</sub>C carbide were detected in all the specimens. Very weak diffraction peaks of Cr<sub>23</sub>C<sub>6</sub> carbide were also identified in the aged specimens of 17 and 20 W alloys. According to a previous study [1–3], tungsten solid solution and carbides are primary strengthening phases in the high-tungsten cobalt-base superalloys.

### 3.2. Effects of high-temperature ageing on creep-rupture properties

Fig. 2 shows the effects of high-temperature ageing on

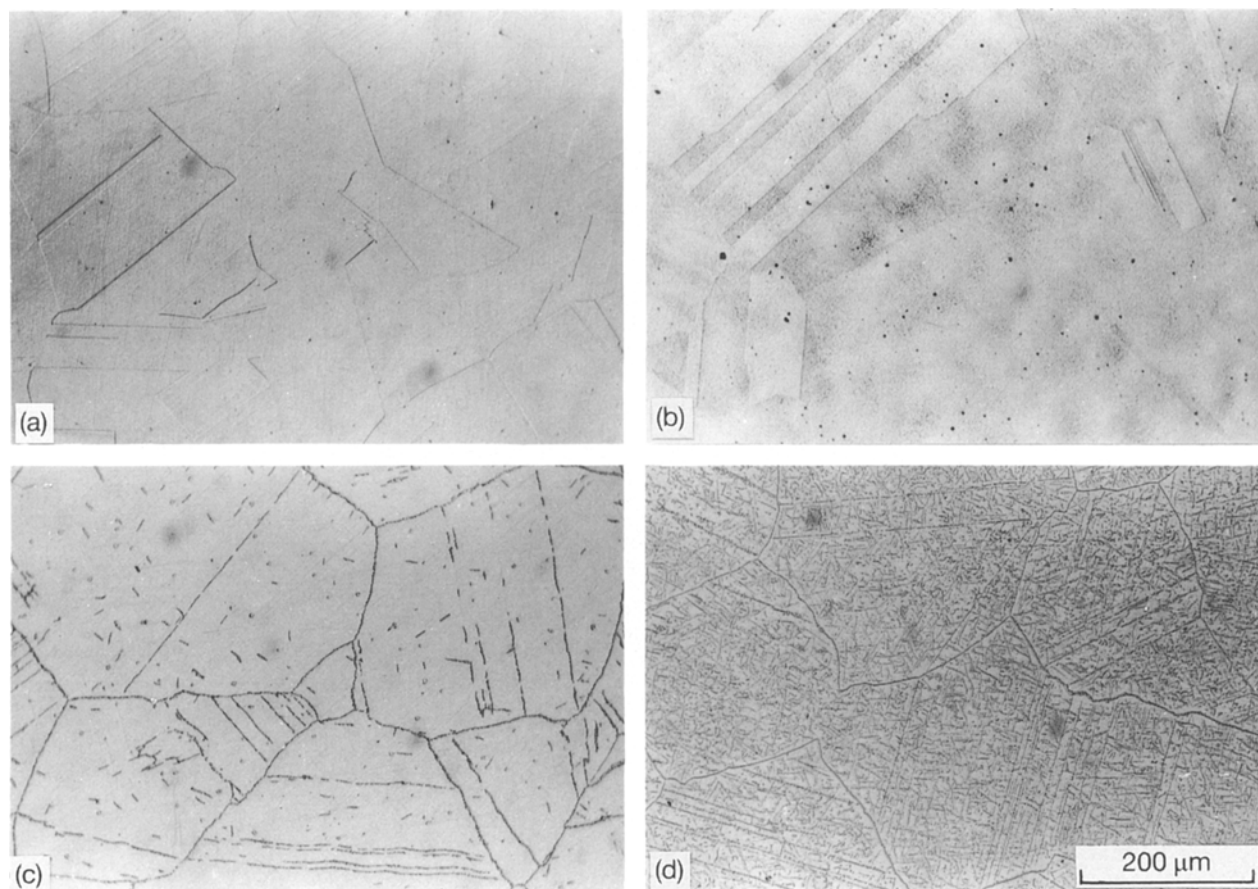


Figure 1 Optical micrographs of the heat-treated specimens in the cobalt-base superalloys. (a,c) 14 W alloy, (b,d) 20 W alloy. (a,b) Solution-treated specimens, (c,d) Specimens aged for 1080 ks at 1273 K.

TABLE III X-ray diffraction data of solution-treated and aged specimens in cobalt-base superalloys

14 W alloy		17 W alloy		20 W alloy		$\beta$ -cobalt*		Tungsten (W)*		$M_6O^*$		$Cr_{23}C_8^*$	
$d_{ob}$ (nm)	I	$d_{ob}$ (nm)	I	$d_{ob}$ (nm)	I	$d_{ob}$ (nm)	I	$d_{ob}$ (nm)	I	$d_{ob}$ (nm)	I	$d_{ob}$ (nm)	I
0.2237	vw	0.2235	w	0.2232	w			0.2238	100				
		0.2166	w	0.2165	w							0.217	50
0.2098	vw	0.2097	vw	0.2099	vw					0.2099	100		
0.2060	vs	0.2058	vs	0.2059	vs	0.20467	100					0.205	100
0.1927	vw	0.1931	vw	0.1929	vw					0.1924	80		
0.1790	s	0.1787	s	0.1787	s	0.17723	40						
0.1579	vw	0.1579	vw	0.1578	vw			0.1582	15				
0.1420	vw	0.1417	vw	0.1421	vw					0.1413	50		
0.1291	w	0.1287	vw	0.1290	vw			0.1292	23				
0.1281	w	0.1282	vw	0.1280	vw					0.1280	80		
0.1264	s	0.1262	s	0.1262	s	0.12532	25						
		0.1254	w	0.1256	w							0.1256	100
0.1091	vw	0.1092	vw	0.1091	vw					0.1090	65		
0.1079	s	0.1078	s	0.1078	s	0.10688	30						
		0.08455	vw	0.08457	vw			0.08459	18				

$d_{ob}$ , observed interplanar spacing; I, relative intensity: w, weak; s, strong; vw, very weak, vs, very strong.

\*X-ray diffraction data of ASTM cards.

the rupture life in the cobalt-base superalloys at 1089 K. High-temperature ageing for 1080 ks at 1273 K improved the rupture life in these alloys, especially under the lower stresses, and the rupture life increased with increasing tungsten content in the aged specimens, while it was almost the same for the solution-treated specimens. The rupture life of the aged specimens was, about 2–5 times longer than that of the solution-treated specimen in the 20 W alloy. As

shown in Section 2, the matrix hardness of the aged specimens increased with increasing of W content, owing to the matrix precipitates of W solid-solution and carbide phases. This led to a large improvement in the rupture life of the alloys containing a larger amount of W. Fig. 3 shows the effects of high-temperature ageing on the rupture life in the same alloys at 1311 K. The high-temperature ageing largely increased the rupture life under the higher stresses at

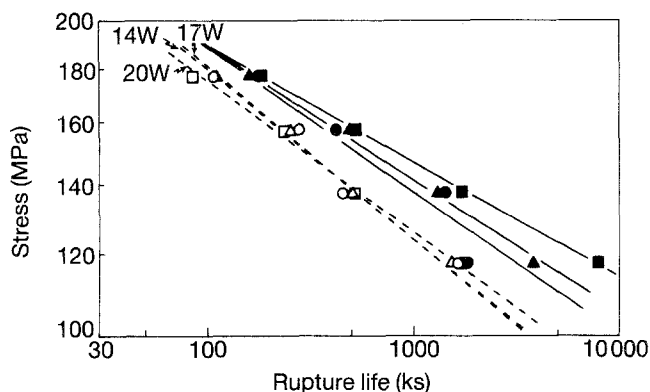


Figure 2 Effects of high-temperature ageing on rupture life in cobalt-base superalloys at 1089 K. Open symbols, non-aged; closed symbols, aged. Alloys, circle 14, triangle 17, square 20 W.

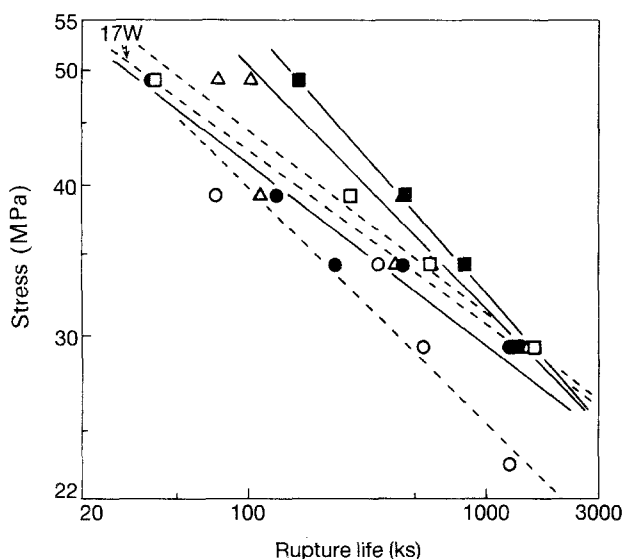


Figure 3 Effects of high-temperature ageing on rupture life in cobalt-base superalloys at 1311 K. Open symbols, non-aged; closed symbols, aged. Alloys, circle 14, triangle 17, square 20 W.

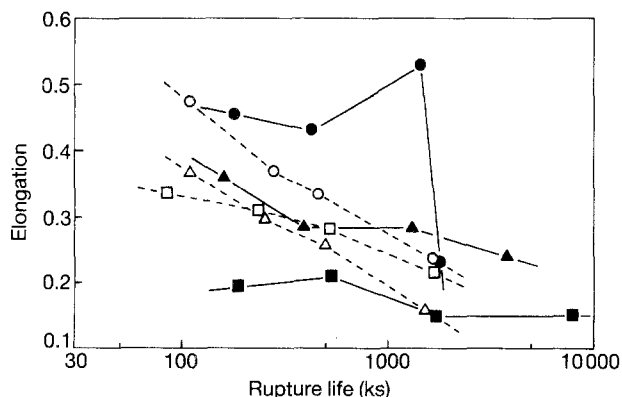


Figure 4 Effects of high-temperature ageing on rupture elongation in cobalt-base superalloys at 1089 K. Open symbols, non-aged; closed symbols, aged. Alloys, circle 14, triangle 17, square 20 W.

1311 K, and the rupture life in both non-aged and aged specimens increased with increasing W content, while the effects of the high-temperature ageing were relatively small in the 17 and 20 W alloys under the lower stresses. These results suggest a large occurrence of W solid solution and carbide phases, which contribute to the precipitation hardening in the solution

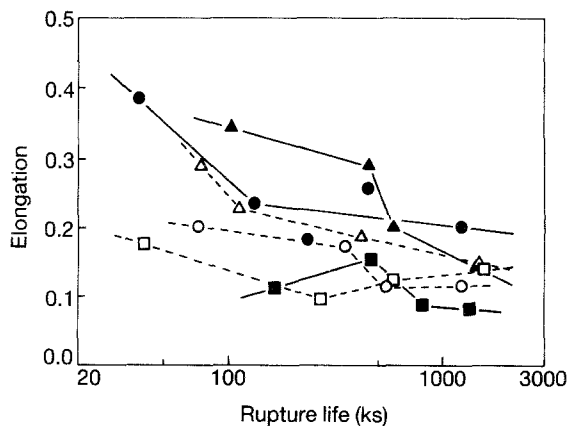


Figure 5 Effects of high-temperature ageing on rupture elongation in cobalt-base superalloys at 1311 K. Open symbols, non-aged; closed symbols, aged. Alloys, circle 14, triangle 17, square 20 W.

treated specimens of the alloys with higher W content during long-term creep [3].

Fig. 4 shows the effects of high-temperature ageing on the rupture elongation in the cobalt-base superalloys at 1089 K. High-temperature ageing slightly increased the rupture elongation in the 14 and 17 W alloys, while it somewhat decreased the creep ductility in the 20 W alloy under higher stresses. The creep ductility seems to decrease with increasing W content in both non-aged and aged specimens of the alloys at 1089 K. Fig. 5 shows the effects of high-temperature ageing on rupture elongation in the same alloys at 1311 K. The creep ductility is not significantly decreased by ageing in these alloys. The creep ductility does not always decrease with increasing W content in both solution-treated and aged specimens, whereas the 20 W alloy exhibits the lowest creep ductility in most cases.

Figs 6 and 7 show the stress dependence of the minimum creep rate in the solution-treated and aged specimens of cobalt-base superalloys at 1089 and 1311 K. The minimum creep rate decreased with increasing W content in the aged specimens of the alloys, especially under lower stresses at 1089 K, although it was almost the same in the solution-treated specimens of these alloys and was proportional to about an eighth of the power of the creep stress at 1089 K [3]. High-temperature ageing also decreased the minimum creep rate at 1311 K, but the effects were relatively small compared with the results at 1089 K. The minimum creep rate decreased with increasing W content in both non-aged and aged specimens in these alloys at 1311 K.

### 3.3. Grain-boundary crack initiation during creep

Fig. 8 shows examples of the creep curves in the cobalt-base superalloys tested under a stress of 137 MPa at 1089 K. The creep curves exhibit long transient creep and an accelerated creep regime, and a relatively short steady-state creep regime in all the specimens. The high-temperature ageing leads to an increase of the rupture life in both the 14 and 20 W alloys, whereas it decreases the creep ductility in the

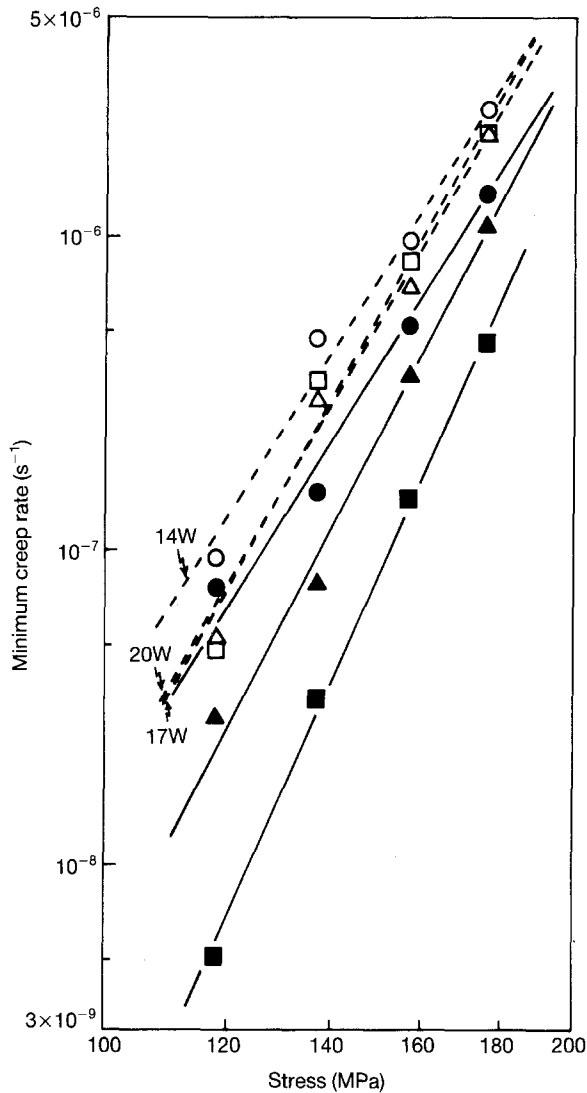


Figure 6 Stress dependence of the minimum creep rate of aged and non-aged specimens in cobalt-base superalloys crept at 1089 K. Open symbols, non-aged; closed symbols, aged. Alloys, circle 14, triangle 17, square 20 W.

20 W alloy. It is interesting to note that the specimens with serrated grain boundaries do not always have the longest rupture life. The arrows shown in the figure indicate the initiation of the grain-boundary cracks on the specimen surface during creep. The grain-boundary cracks initiated in the early stage of creep and at small strains (about 0.01) in the non-aged specimens of the 14 and 20 W alloys, and in the solution-treated and aged specimen of the 14 W alloy, whereas the cracks were formed at larger strains (about 0.03) in the solution-treated and aged specimen of the 20 W alloy and in the aged specimens with serrated grain boundaries of the 14 and 20 W alloys. Similar results to the 20 W alloy were obtained on the 17 W alloy. As shown in the previous study [2], the initiation of the grain-boundary cracks was retarded by serrated grain boundaries in this kind of alloy. The fractal dimension of the grain boundary in the aged specimen was 1.090, comparable with that in the aged specimen with serrated grain boundaries (1.123) in the 20 W alloy, whereas it was 1.033 (a little smaller than the value of the aged specimen with serrated grain boundaries, 1.063) in the 14 W alloy. Thus, the grain-boundary serration caused by high-temperature age-

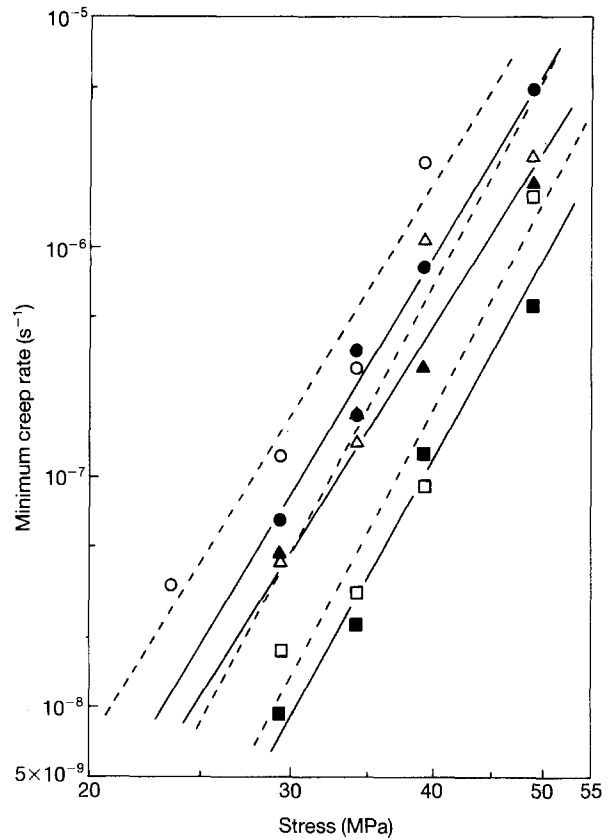


Figure 7 Stress dependence of the minimum creep rate of aged and non-aged specimens in cobalt-base superalloys crept at 1311 K. Open symbols, non-aged; closed symbols, aged. Alloys, circle 14, triangle 17, square 20 W.

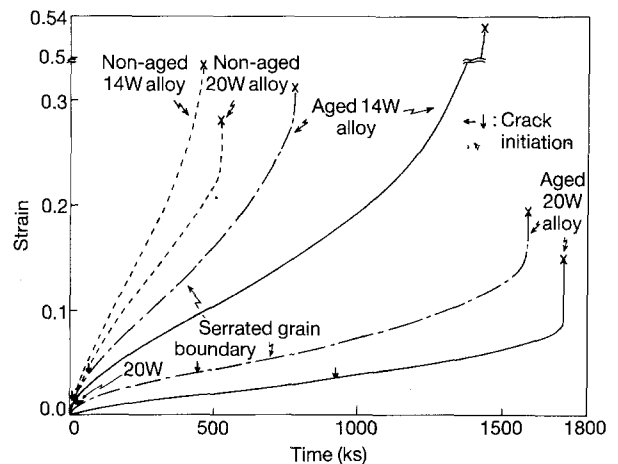


Figure 8 Examples of creep curves in the cobalt-base superalloys tested under a stress of 137 MPa at 1089 K.

ing retarded the initiation of grain-boundary cracks in the cobalt-base alloys with higher W content during creep at 1089 K.

Fig. 9 shows the creep curves of specimens in the cobalt-base superalloys tested under a stress of 29.4 MPa at 1311 K. All the specimens exhibit the creep deformation process similar to that observed at 1089 K. The initiation of the grain-boundary cracks was also retarded by serrated grain boundaries at 1311 K. High-temperature ageing leads to an increase of the rupture life in the 14 W alloy, whereas it is not effective in the 20 W alloy under the lower stresses. A large extent of grain-boundary serration was also

observed in non-aged specimens of the alloys with the higher W content, owing to the precipitation of W solid solution and carbide phases during creep at 1311 K. This is the reason that the high-temperature

ageing is not effective in these alloys. The initiation of grain-boundary cracks occurred at relatively large strain (more than about 0.02) in these alloys except in the non-aged specimen of the 14 W alloy. Thus, in addition to precipitation hardening, serrated grain boundaries which were developed by either high-temperature ageing or precipitation during creep also contributed to strengthening in the cobalt-base alloys with higher W content.

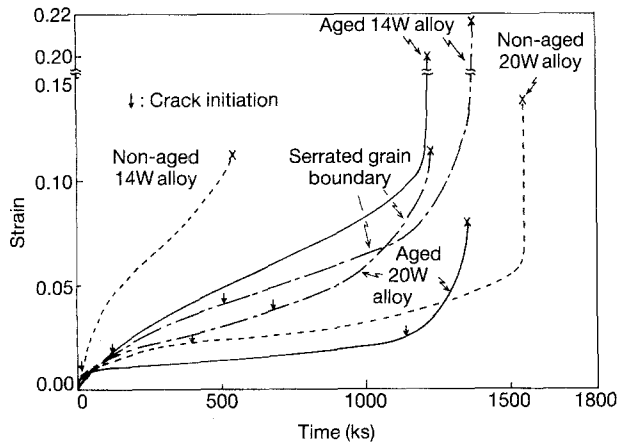


Figure 9 Examples of creep curves in the cobalt-base superalloys tested under a stress of 29.4 MPa at 1311 K.

### 3.4. Microstructures and fracture surface of the ruptured specimens

As revealed in a previous study [2], a ductile grain-boundary fracture occurred in specimens of the L-605 alloy solution-treated and aged for 1080 ks at 1273 K, as well as in specimens with serrated grain boundaries. Fig. 10 shows the microstructures and fracture surfaces in ruptured specimens of the 14 W alloy crept under a stress of 137 MPa at 1089 K. The tensile direction is vertical on the optical micrographs. A few grain-boundary microcracks can be seen near the

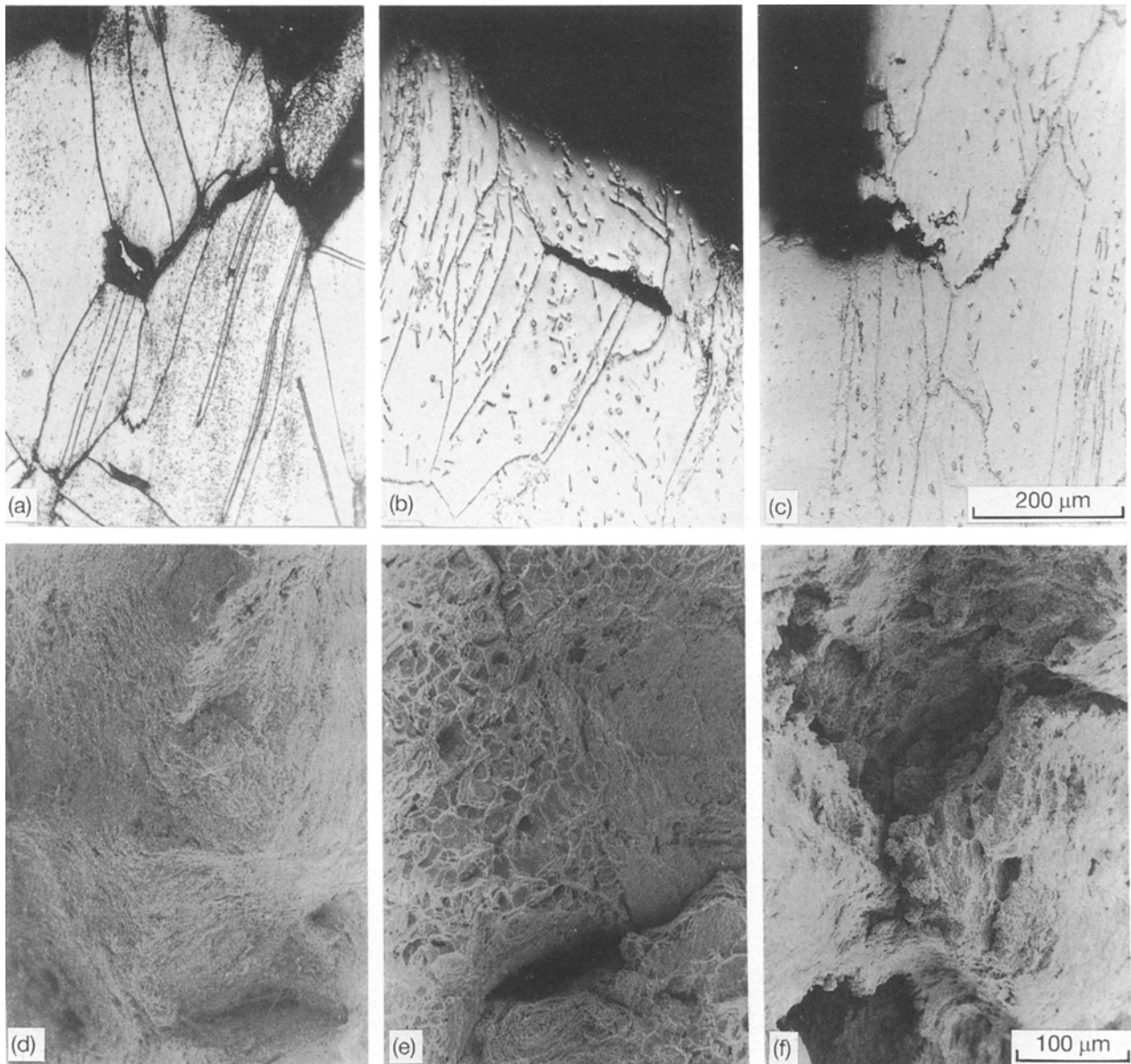


Figure 10 Microstructures and fracture surfaces of the ruptured specimens of the 14 W alloy crept under a stress of 137 MPa at 1089 K. (a,d) Non-aged specimen; (b,e) aged specimen; (c,f) aged specimen with serrated grain boundaries.

fracture surface in the solution-treated and aged specimen (Fig. 10b) as well as in the aged specimen with serrated grain boundaries (Fig. 10c), while a long grain-boundary crack is visible in the non-aged specimen (Fig. 10a). Coarse precipitates of W solid solution and carbide phase can be seen on the grain boundaries and in the matrix of the high-temperature aged specimens (Figs 10b and c), whereas the finer precipitates in the non-aged specimen were Laves phase and  $M_6C$  and  $Cr_{23}C_6$  carbide phases which precipitated during creep at 1089 K (Fig. 10a) [1, 3]. The ductile grain-boundary fracture which is associated with large grain-boundary precipitates can be seen in the solution-treated and aged specimen (Fig. 10e) and in the aged specimen with serrated grain boundaries (Fig. 10f), while the brittle grain-boundary facet is visible in the non-aged specimen (Fig. 10d). Similar results were obtained for the 17 and 20 W alloys.

Fig. 11 shows the microstructure and fracture surface in aged and non-aged specimens of 14 and 20 W alloys ruptured under a stress of 29.4 MPa at 1311 K. The tensile direction is vertical on the optical micrographs. Grain-boundary microcracks can be seen near the fracture surface in these specimens (Fig. 11a–d). Large precipitates of W solid-solution and carbide phases which precipitated during creep are also visible on the grain boundaries and in the matrix in the non-aged specimens (Fig. 11a and c) [3, 5]. Grain boundaries of the non-aged specimens were also serrated, as well as those of the aged specimens, especially in the

alloys with the higher W content. For example, the fractal dimension of the grain boundary in the non-aged specimens was 1.041 for the 14 W alloy, but 1.110 for the 20 W alloy. The latter value is comparable with the fractal dimension of the grain boundary in the aged specimen with serrated grain boundaries of the 20 W alloy (1.123). Ductile grain-boundary fracture surfaces which are composed of dimples and grain-boundary ledges can be observed in aged specimens of the 14 and 20 W alloys ruptured at 1311 K (Fig. 11f and h). Similar results were obtained on the 17 W alloy. The non-aged specimens of the 14, 17 and 20 W alloys showed brittle grain-boundary facets, probably because of severe oxidation at this temperature [2, 3], but small dimples and ledges were also observed in these non-aged specimens [3, 5], although less clearly seen in the micrographs presented here (Fig. 11e and g).

### 3.5. Effects of tungsten content on rupture strength

Fig. 12 shows the effects of tungsten (W) content on the creep-rupture strength at 1089 K in the cobalt-base superalloys. Under non-aged conditions, the rupture strength is not improved by an increase in W content. High-temperature ageing largely increased the rupture strength at longer rupture lives (at 3600 ks) in the alloys containing a larger amount of W, whereas the aged specimens with serrated grain boundaries did

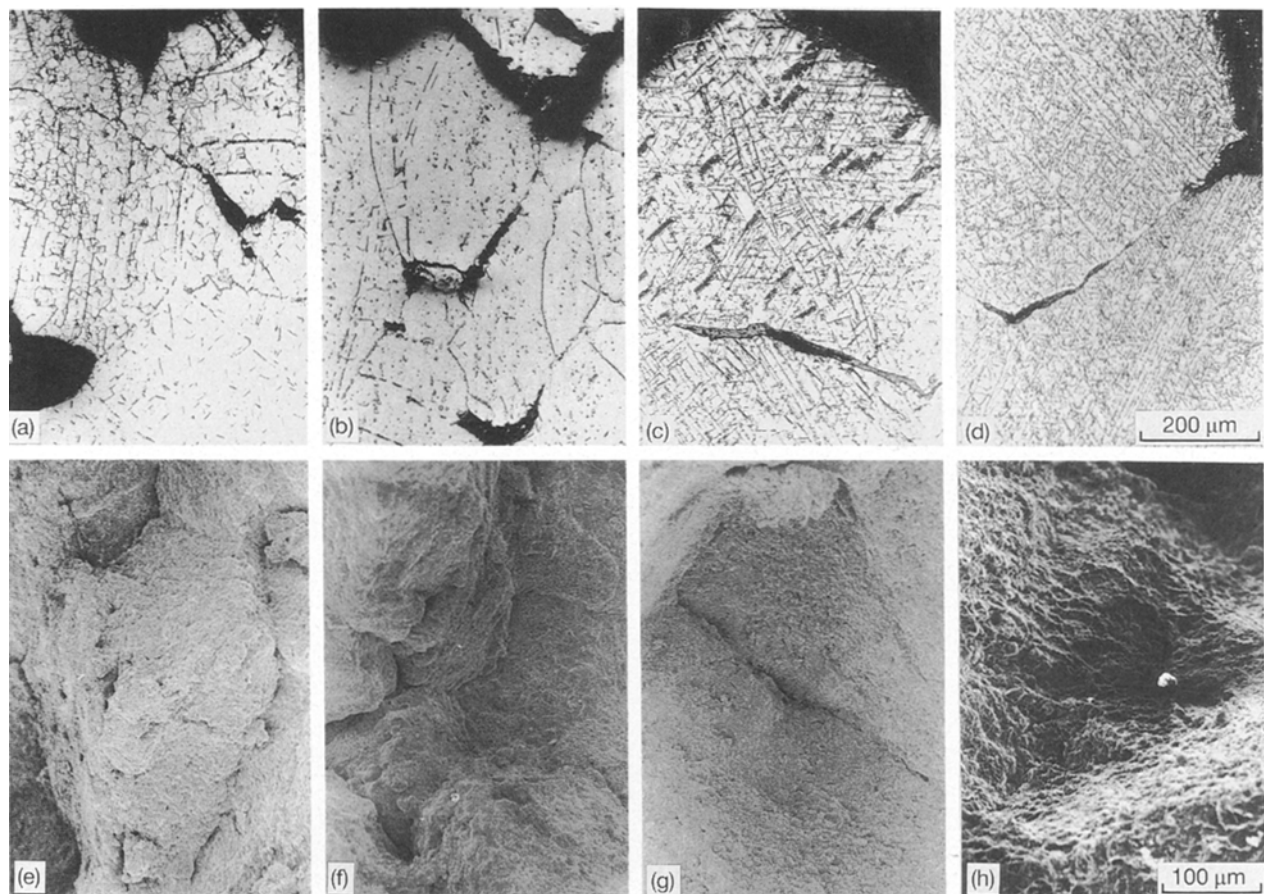


Figure 11 Examples of microstructures and fracture surfaces of ruptured specimens in cobalt-base super alloys crept under a stress of 29.4 MPa at 1311 K. (a,b,e,f) 14 W alloy, (c,d,g,h) 20 W alloy. (a,c,e,g) Non-aged specimen, (b,d,f,h) aged specimen.

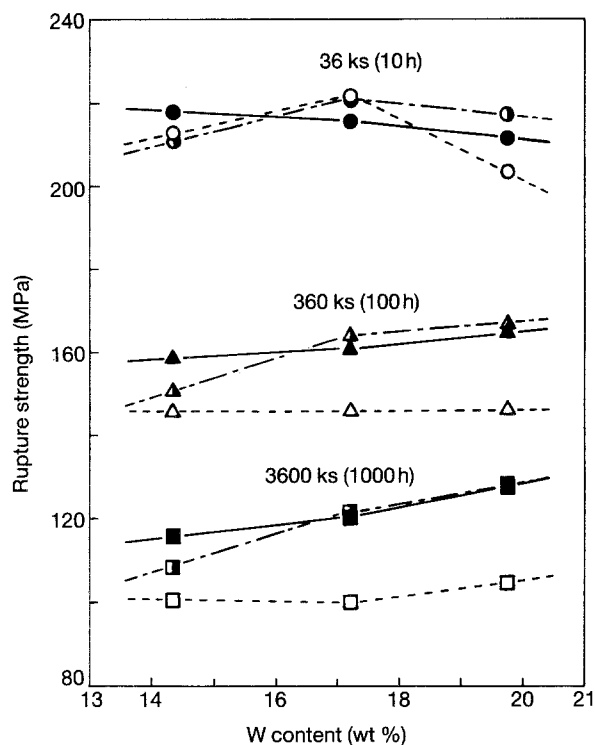


Figure 12 Effects of tungsten content on the creep-rupture strength at 1089 K in the cobalt-base superalloys. (—) Aged for 1080 ks at 1273 K; (---) non-aged; (- - -) aged for 1080 ks at 1273 K, serrated grain boundary.

not always have the highest rupture strength. Fig. 13 shows the effects of tungsten content on the creep-rupture strength at 1311 K in the same alloys. The rupture strength tended to increase with an increase of W content in both aged and non-aged specimens in these alloys. The high-temperature ageing largely increased the rupture strength at the shorter rupture lives (at 36 ks) in the alloys with the higher W content. The solution-treated and aged specimens had almost the same rupture strength as the aged specimens with serrated grain boundaries in these alloys at 1311 K.

#### 4. Conclusions

The effects of high-temperature ageing on creep-rupture properties were investigated using the cobalt-base superalloys containing about 14–20 wt % tungsten. The results obtained are summarized as follows.

1. High-temperature ageing for 1080 ks at 1273 K after solution treatment caused grain-boundary and matrix precipitates of W solid-solution and carbide phases, which resulted in strengthening of grain boundaries and precipitation hardening of the matrix in these alloys. Grain boundaries were serrated owing to the grain-boundary precipitates, especially in the alloys with higher W content.

2. High-temperature ageing largely improved the rupture life, especially in alloys with a higher W content under lower stresses at 1089 K, whereas it somewhat decreased the creep ductility of the 20 W alloy under higher stresses. High-temperature ageing also improved the rupture life without decreasing the creep ductility under the higher stresses at 1311 K, but was less effective under lower stresses at 1311 K.

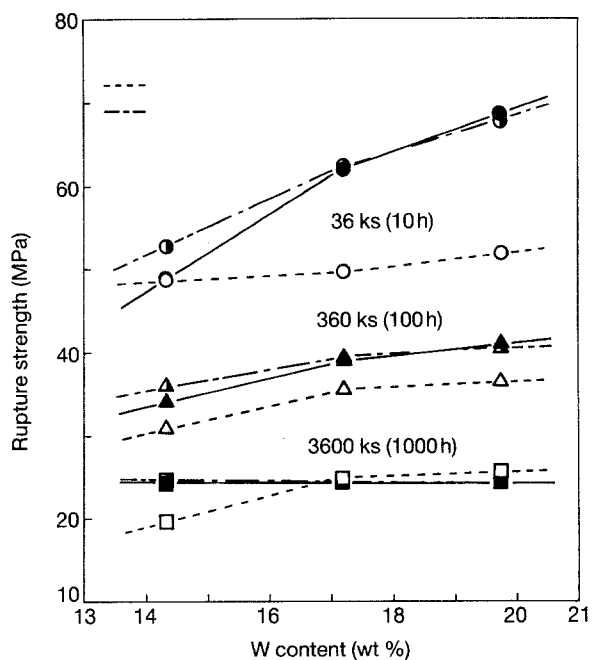


Figure 13 Effects of tungsten content on the creep-rupture strength at 1311 K in the cobalt-base superalloys. (—) Aged for 1080 ks at 1273 K; (---) non-aged; (- - -) aged for 1080 ks at 1273 K, serrated grain boundary.

3. The solution-treated and aged specimens had almost the same rupture strength as the aged specimens with serrated grain boundaries at both 1089 and 1311 K. The rupture strength of the former specimens largely increased with increasing W content under the lower stresses at 1089 K and under the higher stresses at 1311 K.

4. The initiation of grain-boundary cracks was retarded by high-temperature ageing in the alloys with higher W content. The strain-to-crack initiation was larger in aged and non-aged specimens of the alloys with higher W content at 1089 K, while it was almost the same in the aged specimens with serrated grain boundaries of these alloys. A large amount of grain-boundary serration also occurred in non-aged specimens of the alloys with higher W content during creep at 1311 K, and contributed to the retarding of the grain-boundary crack initiation in these alloys.

5. A ductile grain-boundary fracture surface, which was composed of dimples and ledges associated with grain-boundary precipitates, was observed in the solution-treated and aged specimens, as well as in the aged specimens with serrated grain boundaries in these alloys at both 1089 and 1311 K. The fracture surface of the non-aged specimens was a brittle grain-boundary facet at 1089 K, but it became a ductile grain-boundary fracture, as grain-boundary serration occurred owing to grain-boundary precipitates of W solid-solution and carbide phases occurring during creep at 1311 K.

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