THE EFFECT OF STRESS AND TEMPERATURE
ON THE EXTENT OF PRIMARY CREEP IN DIRECTIONALLY
SOLIDIFIED NICKEL-BASE SUPERALLOYS

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Summary

The primary creep strain (PCS) of most directionally solidified nickel-base superalloys at intermediate temperature of 700-800°C and high stress of 490-784 MN/m² is evidently higher than that of conventionally cast alloys. That is due to the shearing of the γ' particles and the large gliding distance of dislocations. PCS increases with increasing stress. The longitudinal PCS of DSK3 at intermediate temperature is quite low, similar to that of the conventionally cast CCK3 alloy. Directional solidification does not increase the longitudinal PCS of K3 alloy at intermediate temperature and high stress. This is due to the creep mechanism of dislocation climbing-over γ' particles. PCS increases slightly with increasing creep stress. The transverse PCS of the directionally solidified alloys is the same of the corresponding conventionally cast alloys. The larger the degree of γ - γ' lattice misfit and the higher the strength, the lower the longitudinal PCS of directionally solidified alloys. At high temperature of 850-980°C and low stress of 196-343 MN/m², the PCS of DS nickel-base superalloy is similar to that of conventionally cast alloys. At high temperature, the PCS of all alloys increases slightly with the increasing creep stress.

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Introduction

Directional solidification of nickel-base superalloys into columnar-grained and single crystal forms results in a marked improvement in creep-rupture life and ductility compared to conventionally cast superalloys. An additional feature of the creep of some directionally solidified nickel-base superalloys, e.g., DSMAR-M200 (1, 2), DSK9 (3), DSK17G (4), etc., is the presence of relatively large primary creep strains at high stress at temperatures around 760°C. In contrast, there is no such primary creep phenomena in some other columnar-grained superalloys, e.g., DSK3 (5). Its primary creep strains are as small as conventionally cast superalloy's. The purpose of this work is to illustrate the difference of temperature and stress dependence of primary creep strain and the mode of primary creep of two kinds of DS superalloys and show the reasons for large primary creep strains.

Experimental Procedures

DSK3, DSK5, DSK9, DS412H, DSK17GW, DS002 directionally solidified nickel-base superalloys with more than 0.6 volume fraction of γ' particles were used in this study. Table 1 shows the chemical composition of DS alloys.

Table I. Chemical Analysis of DS Alloys, Wt%*

Alloy	C	Cr	Co	W	Mo	Al	Ti	Ta	Nb	V	Hf	Ni
DSK3	0.10	10.36	5.10	5.05	4.12	5.59	2.88					Bal
DSK9	0.11	8.10	10.15		6.07	5.96	0.99	4.31				Bal
DSK5	0.10	10.50	10.00	4.85	3.85	5.50	2.50					
DSK17GW	0.175	9.00	10.00	3.70	3.00	5.25	4.40			0.75		Bal
DS002	0.12	8.78	10.24	10.00	0.38	5.54	1.32	2.64			1.55	Bal
DS412H	0.09	5.79	11.86	9.75	2.02	5.26	1.24		2.53		1.45	Bal

*All alloys contain Zr 0.05% and B 0.016% except DS412H which contains B 0.0075%.

Alloys were directionally solidified to blocks by HRS method in a vacuum induction melting furnace. The specimens for creep rupture tests were cut from the lower part of the blocks with standard heat treatment of 1210°C/4 hr + 870°C/32 hr. The axes of longitudinal specimens are parallel to the growth direction of columnar grains, while those of transverse specimens are perpendicular to the growth direction.

Creep rupture test was carried out at 760°C under 689.2 MN/m² for all alloys on the BN-2 creep rupture machine with a device to measure the creep elongation. The corresponding creep curves, the primary creep strain, the rate of secondary creep and the rupture stress are determined respectively. Two typical DS alloys of DSK3 and DSK9 with different primary behaviour were tested at temperature from 700°C to 980°C and at stresses from 196 to 804

MN/m² so that the different dependence of the extent of primary creep in these two alloys on stress and temperature were illustrated and compared with that of conventionally cast alloys.

In order to study the dislocation substructure and the interaction between dislocations and γ' particles produced by creep, creep tests were interrupted after various amounts of deformation at primary and secondary creep stage and the specimens were quenched by forced air to room temperature under loading. It was shown that the dislocation substructure during creep could be frozen in this way. The foils were then cut from the middle part of the specimen by spark cutting and prethinned down to 0.1 mm by lapping. They were finally electropolished by the double jet method in an alcohol solution of 10% perchloric acid at -20°C under 40V (4). Prepared foils were examined in a H700H electron microscope operated at 200KV.

Results

1. Creep and stress rupture behaviour of DS superalloys at intermediate temperature and high stress

Table 2 summarizes the creep and stress rupture data for longitudinal specimens of all DS superalloys including primary creep strain (ϵ_p), secondary (steady-state) creep rate ($\dot{\epsilon}_s$), stress rupture life (t_f) and stress rupture ductility (δ, ψ) at 760°C under 689.2 MN/m², and rupture strength at 760°C for 100 hr (σ_{100}^{760}). Their typical creep curves are shown in Fig.1.

Table II. Creep and Stress Rupture Data for Longitudinal Specimens of DS Alloys

Alloy	$\epsilon_p, \%$	$\dot{\epsilon}_s \times 10^{-2}, \%/hr$	t_f, hr	$\delta, \%$	$\psi, \%$	σ_{100}^{760}
DSK9	4.43	4.49	143.42	14.42	13.84	706.1
DS412H	4.30	2.65	290.42	15.68	17.87	725.7
DSK5	2.10	1.22	545.08	15.40	19.40	755.1
DS002	2.04	1.58	449.38	11.32	12.83	755.1
DSK17GW	1.60	1.44	404.27	15.15	19.15	745.3
DSK3	0.38	0.010	635.00	12.70	17.14	774.8-784.6

It is shown that the primary creep strain is closely related to the creep strength of the alloy. The lower the creep strength of the alloy, the higher the primary creep strain.

2. The effects of stress, temperature and direction of specimen axis on the primary creep strain

The effects of stress at 760°C on primary creep strain, ϵ_p of longitudinal and transverse specimens of directionally solidified DSK3 and DSK9 and that of conventionally cast CCK3 and CCK9 are shown in Fig.2. ϵ_p of longitudinal DSK3, transverse DSK3 and CCK3 increases slightly with increasing stress and are roughly the same with their highest value of 0.7%.

Figure 1 - Typical creep curves for longitudinal specimens of DS alloys at 760°C and 689.2 MN/m²

Figure 2 - The relations between stress and the ϵ_p of directionally solidified DSK9, DSK3 and conventionally cast CCK9, CCK3 alloys at 760°C.

The primary creep strain of longitudinal DSK9 increases remarkably with the increase of stress and it increases from 1.2% to 5% when stress increases from 537.4 MN/m² to 723.5 MN/m². However, the increase of ϵ_p for transverse DSK9 and CCK9 with increasing stress are not so obvious as that of longitudinal DSK9. It can be concluded from Fig.3 that the primary creep behaviour of DSK9 is quite different from that of DSK3. The directional solidification can greatly increase the longitudinal primary creep strain of DSK9 at intermediate temperature and high stress, while its transverse primary creep strain remains similar to that of conventionally cast alloy.

Figure 3 - Typical creep curves for DSK9, CCK9, DSK3 and CCK3 at 760°C and 647.3 MN/m².

Figure 4 - The relations between temperature and ϵ of DSK3 alloy.

In Fig.4, the effect of temperature (850° - 980°C) on ϵ_p of DSK3 at a stress of 245.2 MN/m² is illustrated. When temperature increases from 850°C to 980°C, the increase of the value of ϵ_p is small. The experiment results prove that at high temperatures and under low stress, ϵ_p of DSK9, CCK9, DSK3 and CCK3 are basically the same. Directional solidification does not increase the longitudinal primary creep strain in nickel-base superalloys at high temperature and low stress.

At high temperature of 900°C, the effect of stress on the longitudinal PCS of DSK9 and DSK3 and the PCS of CCK9 and CCK3 are the same. Their ϵ_p increase only slightly with increasing stress (Fig.5).

3. Dislocation sub-structure at primary creep stage

Transmission electron microscope (TEM) observations were conducted to DSK3 at 723.5 MN/m² or 764.9 MN/m² and DSK9 at 647.3 MN/m² after 0.10, 0.20, 2.0 and 5.0 hr. creep test (all at primary creep stage) respectively. The same observations were also conducted to some CCK3 and CCK9 specimens.

Direct observations reveal that there exists obvious δ -fringe appearing at γ - γ' interface in alloy DSK3. The fringes are caused by the tetragonal distortion in matrix at γ - γ' interface, which is due to the γ - γ' lattice constant difference (i.e., the lattice misfit). This means that the γ - γ' lattice misfit in DSK3 alloy is great. Fig.7 and Fig.8 show the

Figure 5 - Relations between stress and longitudinal ϵ_p of DSK9 and DSK3 and that of CCK9 and CCK3 alloys at 900°C.

electron diffraction images of the specimens of DSK3 after 2 hrs creep at stresses of 723.5 MN/m² and 764.9 MN/m² respectively. The following phenomena can be seen in Fig.7 and Fig.8 that in the primary creep process at intermediate temperature and high stress, dislocation can glide relatively freely in matrix γ between the γ' particles. There exist two or more than two different slip systems of $\frac{1}{2}\langle 110 \rangle$ types of dislocation and reactions take place between them. Therefore, a number of 3-dimensional dislocation network can be observed in matrix γ and at γ - γ' boundary. However, since no dislocation pairs of stacking fault can be found in γ' particles, we may conclude that it is unlikely that dislocation may shear γ' particles in primary creep.

The fact that no δ -fringe is observed at γ - γ' interface in the initial-state specimen of DSK9 means that the lattice misfit of γ - γ' is very small. Not only is the dislocation groups similar to that of DSK3 observed in specimens after 0.10 to 5.0 hr creep at stress of 647.3 MN/m², but stacking fault is discovered in many matrix γ' as reported in references (6) and (7) (Fig.9). It means that at the primary creep stage, dislocations not only glide freely in matrix γ , but shear γ' particles as well.

In Fig.10, the electron diffraction image of CCK3 alloy after 5 hrs creep at 760°C and 723.5 MN/m², shows the large size of γ' particles, the high density of dislocation network at γ - γ' interface, and that there is hardly any observable stacking fault and dislocation pairs in γ' . All this is similar to that of DSK3 alloy.

TEM observations have been made to specimens of DSK3 and DSK9 alloys after 0.10, 0.50, 2.0 and 5.0 hrs creep tests (all the specimens are at the primary creep stage) at 980°C and a stress of 245 MN/m². No dislocation in either case shears the γ' particles. However, it is likely that the dislocations of two groups of Burgers vector $\frac{1}{2}a\langle 110 \rangle$ react, and regular 2-dimensional dislocation network at γ - γ' interface can be observed (Fig.11).

4. Calculation and measurement of lattice misfit

The lattice constant and the lattice misfit of γ and γ' in DSK9, DSK5, DSK17GW and DSK3 alloys are calculated by using the formulas suggested in reference (8), in which the composition of γ and γ' phases is obtained from

Figure 6 - δ -type fringes at γ - γ' of the initial-state DSK3.

Figure 7 - Electron diffraction image of DSK3 after 2 hrs creep test at 760°C and a stress of 723.5 MN/m².

chemical analysis of electrolytic residue and are shown in Table 3.

In order to testify the accuracy of the above calculation, the effective distance, d , which is 494 Å, between the dislocation lines at γ - γ' interface in DSK3 specimen after 5,000 hrs ageing at 850°C is measured. Since the dislocation at γ - γ' interface is the dislocation of $\frac{1}{2}a\langle 110 \rangle$ type with a Burgers vector $|b| = 2.54$ Å, according to reference (9) lattice misfit $\delta = |b|/d$, its lattice misfit is 0.50%, which is similar to 0.47% of DSK3 in Table 3. Table 3 shows that primary creep strain at intermediate temperature and high stress is related to γ - γ' lattice misfit. The greater the lattice misfit, the lower the primary creep strain. Same relation between primary creep strain, ϵ_p and γ - γ' lattice misfit, δ can also be obtained in DSK3 and its modified DS alloys (see Table 4).

Figure 8 - Electron diffraction image of DSK3 after 2 hrs creep test at 760°C and a stress of 764.9 MN/m².

Table III Lattice Constant and Lattice Misfit for γ and γ' in DS Alloys*

Alloy	$a_{\gamma}(\text{\AA})$	$a_{\gamma'}(\text{\AA})$	$a_{\gamma'} - a_{\gamma}(\text{\AA})$	$\delta = \frac{ a_{\gamma'} - a_{\gamma} }{a} \%$	$\epsilon_p \%$
DSK9	3.585	3.582	-0.003	0.08	4.43
DS412H	3.588	3.591	0.003	0.08	4.30
DSK5	3.588	3.580	-0.008	0.22	2.10
DS002	3.592	3.583	-0.009	0.25	2.04
DSK17GW	3.593	3.583	-0.010	0.28	1.60
DSK3	3.598	3.581	-0.017	0.47	0.38

* results obtained from phase calculation based on the alloy composition.

a_{γ} lattice constant of γ . $a_{\gamma'}$ lattice constant of γ' .

δ lattice misfit.

Table IV. Chemical Analyses Wt%, Primary Creep Strain, ϵ_p % and Lattice Misfit % for DSK3 and Its Modified DS Alloys

Alloy	C	Cr	Co	W	Mo	Al	Ti	Hf	Ni	$\epsilon_p \%$	$\delta \%$
DSK3	0.10	10.36	5.10	5.05	4.12	5.59	2.88		Bal	0.38	0.47
M1	0.14	8.98	5.95	5.24	3.53	5.44	2.20	1.30	Bal	1.42	0.23
M2	0.13	9.22	6.12	5.73	3.71	5.72	1.76		Bal	2.31	0.17
M3	0.13	9.38	6.12	5.66	3.73	5.79	1.76		Bal	2.49	0.15

Figure 9 - Electron diffraction image of DSK9 after 5 hrs creep test at 760°C and a stress of 647.3 MN/m².

Figure 10 - Electron diffraction image of CCK3 after 5 hrs creep test at 760°C and a stress of 723.5 MN/m².

Discussion

DSK3 alloy has a different primary creep mechanism at intermediate temperature and high stress from that of DSK9 alloy. Creep in DSK3 alloy is due to the slip of dislocation in matrix. When dislocation meets γ' particles, it climbs over the obstacles instead of shearing them. Judging by the δ -fringe at γ - γ' interface in the electron diffraction images and the measured and calculated γ - γ' lattice misfit, we can see that the γ - γ' lattice misfit of DSK3 alloy is very large. This conclusion is confirmed by the observation of the existence of γ - γ' interface dislocation in long ageing specimens. When the γ' particle grows, the γ - γ' lattice misfit can only be adjusted through forming γ - γ' interface dislocation (11). A high

Figure 11 - Electron diffraction image of DSK3 after 5 hrs creep test at 950°C and a stress of 245.2 MN/m².

misfit causes a high coherent strain field near the γ - γ' interface which prevents dislocation from shearing γ' phase. Since there exist the creep mechanism of dislocation climbing over γ' phase and γ - γ' interface dislocation, and the multiplicity of $\{111\} \langle 110 \rangle$ slip system, the slip distance at the primary creep stage decreases rapidly. The distance is limited to one or a few γ' particle sizes. Therefore, the primary creep strain of DSK3 alloy at intermediate temperature and high stress is very small.

The creep of DSK9 alloy is different from that of DSK3, it creeps when dislocation slips in matrix γ and shears γ' particles it don't fronts. References 1 & 2 point out, since very high energy is needed for $\frac{1}{2}a\langle 110 \rangle$ super lattice dislocation pairs to shear γ' particles in nickel-base superalloys with high volume fraction of γ' phase, the viscous glide shearing (cutting) of γ' particles of intrinsic/extrinsic stacking fault pairs with a Burgers vector of $a\langle 112 \rangle$ takes place. However, such creep deformation is quite heterogeneous. Dislocations can glide large distances without leaving their slipping plane. Steady-state creep is reached only when enough interactions have occurred between $\{111\} \langle 112 \rangle$ slip systems to limit glide distance of dislocation to the scale of one or a few γ' precipitate particles. So, its primary creep strain is large. The primary creep of DSK9 alloy is also of the similar mechanism of dislocation shearing γ' particles. Therefore, DSK9 also has a high primary creep strain. This creep mechanism of DSK9 is related to the relatively small γ - γ' lattice misfit and to the fact that there is a low coherent strain field at the γ - γ' interface.

The fact that the primary creep of transverse specimens in directionally solidified alloy is similar to that of conventionally cast alloys indicates the importance of grain boundary in creep process. Because of the constraint adjacent grains, grain boundary is an obstacle to dislocation slip. Hence, the conventionally cast polycrystal materials of CCK9, CCK3, etc. and the transverse specimens of directionally solidified alloys have a small primary creep strain. X-ray measured orientation of columnar-grain axis of directionally solidified specimen is $\langle 001 \rangle$ (4). Moreover, there exist cubic orientation relation between γ and γ' . Therefore, crystal in

longitudinal creep specimen has an orientation of $\langle 001 \rangle$, i.e., the $\langle 001 \rangle$ oriented columnar grain boundary parallels tensile axis of specimen. This means little obstacle to dislocation glide. So, directionally solidified alloys with a small degree of lattice misfit tends to have a large primary creep strain at intermediate temperature and high stress.

At high temperature of 850°C to 900°C, the primary creep strain of various directionally solidified alloys are very low, similar to that of conventionally cast alloys. The diffraction images indicate that primary creep at high temperature is due to dislocation climbing over of γ' particles in matrix γ . Since it is easy for two groups of $\frac{1}{2}a\langle 110 \rangle$ dislocation to react, homogeneous arrangement of dislocation occurs at lower strain, and the glide distance of dislocation is limited at the beginning of creep. Therefore, directionally solidified alloys have a low primary creep strain as that of conventionally cast alloys.

At intermediate and high temperatures, primary creep strain of directionally solidified alloys increases with increasing stress. Judging from the relations between creep stress and dislocation density in secondary creep stage of DSK3 at intermediate and high temperatures, the dislocation density of nickel-base alloys, as that of other metals, increases with increasing stress. That is one of the reasons for the increase of primary creep strain with increasing creep stress. Besides, larger creep strain is also due to the fact that, in viscous slip process, the glide speed of dislocation increases with increasing stress, and it is less probable for dislocation at high stress to leave its slip plane than at low stress, so that a dislocation will sweep out more slip planes before it stops.

Conclusion

- (1) At intermediate temperature and high stress, the longitudinal primary creep strain of DS nickel-base superalloys is related to the strength of alloys which is related to its lattice misfit of $\gamma-\gamma'$. The greater the strength, the lower the primary creep strain. And the larger $\gamma-\gamma'$ lattice misfit, the stronger the alloy and the smaller the primary creep strain.
- (2) At intermediate temperature and high stress, the primary creep strain of most DS nickel-base superalloys is larger than that of the conventionally cast alloys of the same kind. This is due to the creep mechanism of dislocation shearing of γ' particles.
- (3) The primary creep strain of DSK3 alloy at intermediate temperature and high stress is very low, similar to that of the conventionally cast CCK3 alloy. Directional solidification does not increase the primary creep strain of K3 family alloys at intermediate temperature and high stress. That is due to the creep mechanism of dislocation climbing over the γ' particles.
- (4) At high temperature and low stress, the longitudinal primary creep strain of DS nickel-base superalloys is similar to that of conventionally cast alloys. Directional solidification does not increase the longitudinal primary creep strain of alloys at high temperature and low stress.
- (5) The primary creep strain of DSK3 and transverse DSK9 slightly increases with increasing stress at various temperatures and stresses, while that of longitudinal specimens of DSK9 considerably increases at intermediate temperature and high stress.

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