THE DEVELOPMENT AND CHARACTERIZATION

OF A HIGH PERFORMANCE EXPERIMENTAL SINGLE CRYSTAL SUPERALLOY

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Summary

A titanium-free single crystal alloy designated MXON is shown to possess a substantially higher creep strength at high temperatures than that of the currently available conventional single crystals. A further investigation of the role of cobalt showed that the absence of this element improves the overall creep resistance of the alloy. The degree of chemical homogeneity achieved through the solutioning durations affects the subsequent stability of the alloy during high temperature creep. Small amounts of a tungsten-rich μ phase (0.3%) were found to precipitate if sufficient chemical homogeneity is not achieved prior to the tests. Increasing the cobalt level beyond 5% facilitates the formation of this phase even at intermediate temperatures. The precipitation of the μ phase, which lowers the creep resistance of the alloy, can be avoided by longer solutioning durations prior to the creep tests. The alloy containing 7.5% cobalt shows an anomalously high primary creep due to inhomogeneous {111}<112> slip. The high cycle fatigue strength is not affected by the μ phase. The MXON alloy has excellent corrosion resistance due to the development of a stable oxide layer.

Introduction and Approach

Although the early developmental work on nickel-base single crystal superalloys dates back to the mid 60's, it was only around 1976 that the idea of removing grain boundary strengthening elements paved the way for developing a variety of new compositions showing considerably higher creep potential than that projected by the earlier work. Since that period, much effort has been devoted to both the developmental and the behavioural aspects of single crystal superalloys. The critical stages of some modern aircraft engines are presently being equipped with single cystal blades (1) and further advances in alloy design are expected for future applications. In view of the revived interest in single crystals, a program was initiated at ONERA aimed at developing a more fundamental understanding of the role of various factors which govern their mechanical and environmental behaviour as well as identifying some new high strength alloy compositions. From the standpoint of mechanistic studies the CMSX-2 alloy (2,3) has served as a starting point and it was decided to develop some simple compositional modifications of this alloy with the following objective and guidelines :

- identify a composition whose creep strength at elevated temperatures is significantly higher than that of current conventional single crystals but which is also highly corrosion resistant.

- the cast single crystal alloy should be virtually devoid of primary γ'associated with the γ/γ'eutectic regions which should permit an easy solutioning of the secondary γ'phase within a short period of time.

- the temperature interval between completeγ' solutioning and the incipient melting point (heat treatment window) should be large.

A limited alloy development work performed in conjunction with the microstructural studies of cast structures led to the identification of the following base-line composition which was selected for mechanical property evaluation and corrosion studies:

$$Ni - 5Co - 8Cr - 6Ta - 6.1Al - 2Mo - 8W \text{ (wt. percent)}$$

This alloy, later referred to as MXON, contains no titanium but has appreciable amounts of aluminium, chromium and tantalum. The addition of 2% molybdenum led to an almost total elimination of the eutectic regions and provided a large "heat treatment window". Due to the controversial effects of cobalt on mechanical properties reported in the literature (4) an investigation was also undertaken on two derivatives of the MXON alloy : a cobalt-free alloy (MXON-OCo) and another version containing 7.5% cobalt (MXON - 7.5 Co). This work describes and analyses some of the properties of the MXON alloy and derivatives with the overall objective of developing a better understanding of their mechanical behaviour which may contribute to further advancement in alloy development.

Experimental Details

Dendritic single crystals of MXON alloys were cast in the [001] orientation using the seeded technique at a withdrawal rate of 15 cm/h in a high temperature gradient. The single crystal bars selected for this investigation were within 5° of the [001] direction. The average spacing of primary dendrites was 160 μm and the interdendritic regions contained negligible amount of γ/γ' eutectic in the three alloys. The heat treatment

window of these alloys was found to be about 25°C. Based on our recent work on the effect of heat treatments on creep strength (5) the following heat treatments were applied :

MXON : $1310°C/\frac{1}{2}h$ or 1h or 6h/ A.C. + 1100°C/4h/A.C. + 850°C/24h

MXON - 0Co and 7.5 Co : $1310°C/\frac{1}{2}h$/A.C. + 1100°C/4h/A.C. + 850°C/24h.

For the MXON alloy despite the complete solutioning of the γ' phase within a few minutes the solutioning duration was varied from 1/2h to 6h in order to assess the consequence of a greater chemical homogeneity of the alloy on creep. The tensile creep behaviour of the three alloys was investigated in the temperature range 760 - 1050°C. To determine the possible effects of γ' wt. fraction and its chemical composition the γ' particles were electrolytically extracted according to a standard technique (6). The composition of this phase was determined by electron microprobe analysis of the extracted and cold-compacted particles. The lattice parameter measurements were made on extracted γ' by X-ray diffraction. The identification of a small amount of platelet-like phase (μ phase) appearing in stress rupture specimens was carried out by electron diffraction analysis using a Jeol 200 CX transmission electron microscope (TEM) with energy dispersive X-ray spectroscopy (EDS) capability. To study the dislocation structures, thin foils were cut from the interrupted creep specimens and examined in a Jeol 200 CX microscope operated at 200 KV.

Microstructural Characterisation

The microprobe analysis of segregations across the dendrites shows that except for tungsten, whose concentration in the dendrite core may be as high as 11.5 wt.% , a reasonably good chemical homogeneity is attained for all the other elements only after 30 minutes of solutioning at 1310°C. After 2 hours the central portion of the dendrites still contains around 9.5% of tungsten but after 6 hours of solutioning treatment the maximum concentration is down to about 8.5%. The heat treatments applied to the three alloys resulted in a homogeneous precipitation of well aligned cuboidal precipitates along < 001 > directions without any interfacial dislocations. The weight fraction, the mean size and the lattice parameter of the γ' phase as a function of cobalt concentration are listed in Table I. Repeated determinations showed that cobalt additions did not affect the lattice parameter but led to a slight increase in the weight fraction and a perceptible decrease in the mean particle size. The typical γ' morphology in the MXON alloy after heat treatments is illustrated in Fig. 1. The composition of the extracted γ' phase for the three alloys is very similar leaving aside the cobalt substitution for nickel in the cobalt containing alloys (Table II).

Table I — Microstructural characteristics of the γ' phase

Alloy	Weight fraction (%)	Mean size (Å)	Lattice parameter (Å)
MXON-0Co	66.8	4170	3.5842
MXON	68.8	3610	3.5849
MXON-7.5 Co	69.6	3460	3.5847

Table II — Chemical composition of the γ' phase (wt. %)

Alloy	Ni	Co	Cr	W	Mo	Al	Ta
MXON-OCo	73.02 ± 0.72	—	2.29 ± 0.06	7.68 ± 0.38	0.94 ± 0.09	8.47 ± 0.33	7.61 ± 0.23
MXON	69.49 ± 0.69	3.00 ± 0.09	2.33 ± 0.09	8.38 ± 0.50	1.04 ± 0.07	8.11 ± 0.36	7.65 ± 0.23
MXON-7.5Co	67.89 ± 0.68	4.67 ± 0.14	2.12 ± 0.06	8.18 ± 0.41	1.07 ± 0.10	8.45 ± 0.33	7.62 ± 0.38

Creep and Fatigue Behaviour

Effect of Chemical Homogeneity and Cobalt Content on Creep in the Temperature Range 760–1050°C

The creep properties of the MXON alloy in the fully heat treated condition for three solutioning durations and cobalt concentrations are summarized in Table III. The 1000-hour creep rupture strength of this alloy (solutioned 6 hours) is also compared in Fig. 2 with two modern single crystal alloys, CMSX-2 (5) and Alloy 454 (7) and a nickel-base eutectic, Cotac 744 (8) developed at ONERA during the late 70's. It is interesting to note that the creep strength of MXON is very close to that of the eutectic in the high temperature regime which illustrates the remarkable intrinsic creep potential offered by a titanium-free single crystal alloy. Clearly, the cobalt-free version which will make the alloy still simpler in composition is virtually as strong as the MXON alloy (5%Co). A mere 30-minute solutioning is sufficient to produce the best combination of creep strength in the whole temperature range 760–1050°C for the cobalt-free alloy (Table III). The temperature advantage offered by MXON over the CMSX-2 alloy at 1000°C is about 30°C and 50°C over alloy 454 (PWA 1480).

Table III — Creep results as a function of solutioning duration and cobalt concentration

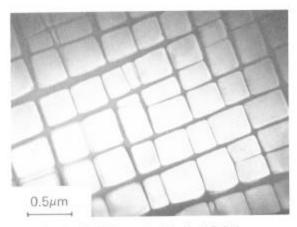| Temperature (°C)	Stress (MPa)	Creep parameters	MXON-OCo Sol. 30 mn	MXON Sol. 30 mn	MXON Sol. 1 h	MXON Sol. 6 h	MXON-7.5Co Sol. 30 min
760	750	1 % creep (h)	330	234	—	—	1
		Rupture (h)	1189	1190	—	—	778
		Elongation (%)	8.9	14.9	—	—	18.7
		Min. creep rate x 10^8 (s^{-1})	0.62	0.91	—	—	1.85
950	240	1 % creep (h)	168	109	142	183	154
		Rupture (h)	513	374	408	531	415
		Elongation (%)	23.6	25.6	25.5	32.3	25.0
		Min. creep rate x 10^8 (s^{-1})	0.86	1.91	1.61	1.17	1.73
1000	200	1 % creep (h)	117	—	118	105	63
		Rupture (h)	248	—	273	265	197
		Elongation (%)	14.0	—	17.5	—	20.4
		Min. creep rate x 10^8 (s^{-1})	1.30	—	1.44	1.59	2.91
1050	140	1 % creep (h)	552	550	685	—	450
		Rupture (h)	671	808	829	—	591
		Elongation (%)	13.7	—	—	—	7.7
		Min. creep rate x 10^8 (s^{-1})	0.17	0.12	0.18	—	0.26

Fig. 1 — T.E.M. superlattice dark field image
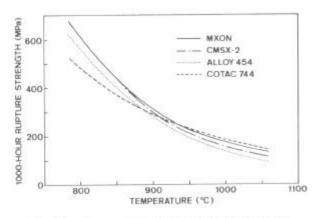of the γ' precipitates in MXON.

Fig. 2 — Comparative creep strengths of single
crystal alloys MXON, CMSX-2, 454 and the D.S.
eutectic COTAC 744.

We will first discuss the creep behaviour of these alloys at 760°C and then at high temperatures. A comparison of the creep behaviour of the fully heat treated alloys (solutioned 30 minutes) at 760°C and 750 MPa shows an increase of both the primary and secondary creep rates with increasing cobalt content (Table III and Fig. 3). A dramatic increase in the extent of primary creep is observed for the alloy containing 7.5% cobalt (Fig. 3) ; the stress rupture life for this alloy is also significantly lower. On the other hand, the elongation to rupture increases with the increasing cobalt concentration. Some tests were interrupted during primary creep in order to examine the corresponding dislocation structures. Typical deformation structures of the cobalt-free and the 7.5% cobalt containing alloys are compared in Fig. 4. The dislocation structure of the former alloy is very similar to that of the MXON alloy (5% Co). This type of homogeneous deformation which involves the glide of a/2 < 110 > type matrix dislocations during the early stages of creep throughout the specimen is responsible for low creep rates. Conversely, the deformation structure in the MXON-7.5 Co alloy is extremely inhomogeneous : the γ' particles are being sheared over long distances by {111} <112> planar slip resulting in the creation of intrinsic/extrinsic stacking faults. This type of deformation is responsible for high primary creep and it retards a uniform strain hardening. These observations unambiguously confirm our recent investigation of the deformation structures associated with the more or less homogeneous nature of deformation in primary creep in another single crystal alloy (3,5). While analysing the creep behaviour of the MXON-7.5Co alloy it should be recalled that the corresponding decrease in γ' size may play some role in facilitating γ'shearing and thereby contributing to inhomogeneous deformation. However, a comparison of the creep behaviour of MXON subjected to two different precipitation heat treatments resulting in two distinct γ' mean sizes (2000 and 3610 Å) shows that although the smaller precipitate size also promotes much higher primary creep rates, the extent of primary creep remains much lower than in the alloy containing 7.5% cobalt (Fig. 5). Hence , in order to account for such a high primary creep extent in this alloy, some other effect of cobalt should be considered. One possibility could be the decrease in stacking fault energy of the matrix. It has been suggested by Leverant and coworkers (9) that elements such as Co, Mo and Cr which partition preferentially to the matrix lower its stacking fault energy and hence may facilitate the formation of a/2 <112> type widely dissociated dislocations which are required for the intrinsic/extrinsic shearing of the γ' particles during creep around 760°C. This is precisely the predominant mechanism operative in MXON-7.5 Co alloy during the early stages of creep as opposed
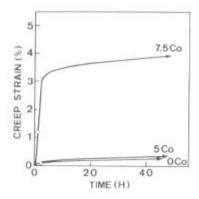
Fig. 3 — Creep behaviour at 760°C and 750 MPa as a function of cobalt concentration. Circles correspond to the microstructures shown in Fig. 4.
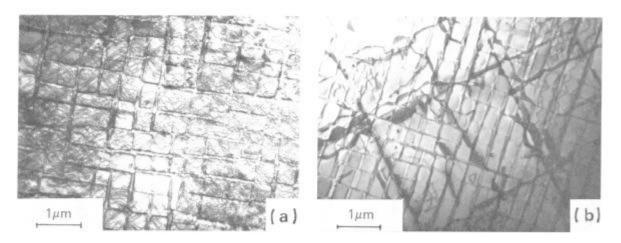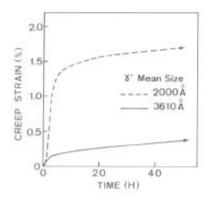

Fig. 4 (a) and (b) — Typical deformation microstructures during primary creep at 760°C and 750 MPa. (a) MXON-O Co, ε = 0.14 % ; (b) MXON-7.5 Co, ε = 1.20 %.
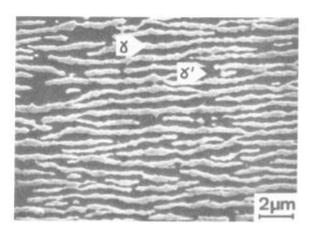

Fig. 5 — Effect of particle size on primary creep of MXON at 760°C and 750 MPa.

Fig. 6 — S.E.M. micrograph showing the rafted γ' structure after 90 h at 1050°C and 140 MPa (ε = 0.52 %). The section is parallel to the stress axis.

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to the situation in the other two alloys where the deformation occurs primarily by the glide of a/2 <110> dislocations in the matrix. The shearing of the precipitates in the cobalt-free and the MXON-5Co alloys occurs by {111}<112> slip only during the late stages of primary creep. The composition of the matrices of MXON alloys calculated by the mass balance technique from γ' analysis and bulk alloy chemistry show that the cobalt concentration in the matrix is increased from about 9.7 to 14 wt.% when the bulk concentration is varied from 5 to 7.5 % . The present experimental evidence therefore strongly suggests that an increase in the cobalt level beyond a certain critical value facilitates easy shearing of the particles by {111}<112> planar slip primarily by lowering the stacking fault energy of the matrix. However, more detailed investigation is in progress to determine the eventual contribution of particle size in the MXON-7.5Co alloy (10).

The prominent structural feature observed during creep of MXON alloys beyond 900°C is the formation of long regular γ' rafts perpendicular to the stress axis (Fig. 6). The formation of such rafts is believed to reduce considerably the secondary creep rate by inhibiting dislocation climb around the γ' phase and thereby imparting a very high creep strength to these alloys. Some recent studies on <001> oriented single crystal superalloys (11,12) have produced convincing evidence that the formation of rafts requires a negative γ/γ' misfit at temperatures where the raft formation is observed. A regular rafted structure was shown to develop during high temperature creep in the CMSX-2 single crystals alloy (3,5). The γ/γ' misfit in this alloy was found to be +1.4 x 10^{-3} at room temperature. The lattice parameter of γ' in MXON was calculated by using the method of Watanabe and Kuno (13) and considering the experimentally determined γ' composition (Table II) ; the value of the parameter thus determined is 3.5847 Å which is remarkably close to that determined by X-ray diffraction (3.5849 Å). The calculated parameter of the matrix was found to be 3.5854 Å ; the γ/γ' misfit is about -2.10^{-4}. Recent work of Grose and Ansell (14) has shown that in a series of γ' strengthened nickel-base alloys the thermal expansion coefficients of the γ' phase are consistently smaller than that of the matrix. One can therefore conclude from the present information that even if the misfit is slightly positive at room temperature it will tend to become negative at high temperatures and hence a rafted γ' morphology would be expected. A negative misfit at room temperature would clearly be increasingly negative when the temperature increases.

In order to evaluate the effect of cobalt content on phase stability, stress rupture specimens crept in the temperature range 760-1050°C were examined metallographically. At 760°C, only the MXON-7.5Co alloy contained a small amount of a platelet like phase localized in the dendrite cores and is thought to be partly responsible for decreasing the stress rupture life. However, the extremely high primary creep rate in this alloy at 760°C is not related to the presence of these platelets since they precipitate at a much later stage. At higher temperatures all the three alloys subjected to a 30-minute solutioning treatment prior to the creep tests contained some amount of this phase. The amount of platelets was found to be maximum at 950°C (about 0.3 volume%) in the 30-minute solutioned MXON and MXON-7.5 Co alloys (Fig. 7b). The cobalt-free version was least prone to the platelet-like phase (fig. 7a). Electron diffraction and X-ray microanalysis identified these platelets to be a tungsten-rich μ phase with a rhombohedral symmetry. The hexagonal lattice parameters determined from electron diffraction analysis were found to be : a = 4.73 Å and c = 25.36 Å. The composition of the μ phase as determined in the MXON alloy is as follows : 13.3 Ni-4.5 Co-10.5 Cr-57 W-14.7 Mo. The localized precipitation of this tungsten-rich phase is a consequence of the tungsten segregations detected in the dendrite
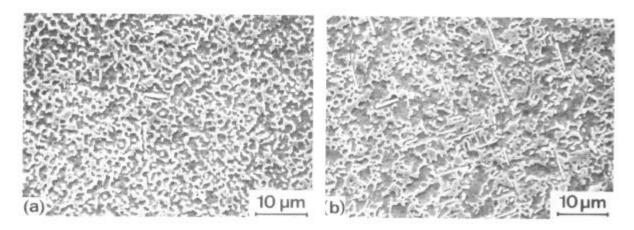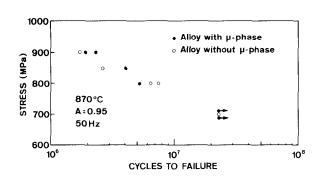
Fig. 7 (a) and (b) — S.E.M. micrographs showing µ-phase precipitation after creep failure at 950°C and 240 MPa. The sections are perpendicular to the stress axis. (a) MXON-0Co ; (b) MXON-7.5 Co.

cores by microprobe analysis. Some specimens of the MXON alloy were solution-treated for 1 hour and 6 hours to reduce the tungsten segregation and subsequently tested in creep at 950°C and 240 MPa (Table III). As expected, the amount of µ phase decreased substantially after one hour of solutioning and only a few isolated platelets could be occasionally detected after the 6-hour solutioning treatment. The improvement in creep properties at 950°C as the solutioning duration is increased seems to be related primarily to the decreasing amount of µ phase, the detrimental effect of this phase arising from the partial depletion of the matrix in W and Mo. A better chemical homogeneity may however be also partly responsible for this improvement. It is also worth noticing that in general, the MXON-7.5 alloy has the highest creep rates whereas the cobalt-free version shows the lowest rates. These observations strongly suggest that even small amounts of the tungsten and molybdenum-rich µ phase do lower the creep strength of the MXON alloys. In conclusion, the present results clearly show that the absence of cobalt improves the creep resistance and that the cobalt-free alloy is least prone to the µ phase precipitation. The 5% cobalt concentration seems to be a limit beyond which the overall creep resistance of these alloys is decreased significantly due to reasons which are not exclusively related to the precipitation of µ phase.

Effect of µ Phase on High Cycle Fatigue

The high cycle fatigue behaviour of MXON alloy was investigated at 870°C in order to evaluate the possible role of µ phase platelets in crack initiation and hence on the fatigue life. The specimens were solution treated for 30 minutes or 6 hours to achieve different degrees of chemical homogeneity ; they were then held for 400 hours at 950°C. In the 30-minute solution treated specimens the amount of µ phase precipitated after 400 hours was comparable to that found in the stress rupture specimens crept at 950°C ; the 6-hour treated samples showed very few isolated particles. The tension-tension fatigue tests were then performed on the hour-glass type specimens and the result are shown in Fig. 8. It is obvious that the presence of µ phase has no effect on the high cycle fatigue behaviour. The examination of the fracture surfaces shows that the crack initiation occured exclusively on micropores in the interior of the specimen. The propagation was found to be frequently of stage 1 type along {111} type planes but in some cases the stage 1 is followed by a stage 2 non-crystallographic type propagation, perpendicular to the stress axis. The 10^7-cycle fatigue limit
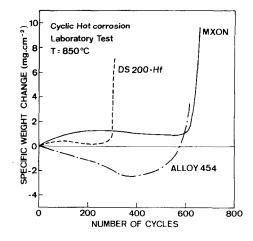
Fig. 8 — Effect of µ-phase on the H.C.F. pro-
perties at 870°C of MXON.

Fig. 9 — Comparative corrosion behaviour of
alloys MXON, 454 and DS 200 + Hf.

of this alloy ($\simeq 700$ MPa) was found to be equivalent to that of the CMSX-2 single crystals obtained under similar solidification conditions (15).

Corrosion Behaviour

The corrosion tests were run at 850°C in air under atmospheric pressure on samples of the uncoated MXON alloy contaminated with Na_2SO_4 (0.5 mg. cm^{-2} renewed every 50 hours). These samples were subjected to a one-hour thermal cycle during the test. The salt level deposited on the specimens corresponds to that typically found in a burner rig using kerosene fuel containing 0.15 per cent sulphur and a 5 ppm NaCl contamination introduced in the air. The samples were weighed every 50 hours in order to follow the evolution of corrosion attack. Comparative tests were performed on DS 200 + Hf and the "454" single crystal alloy. The weight change curves for the three alloys are plotted as a function of time in Fig. 9. These curves show the usual incubation period with a very low corrosion rate followed by a period of accelerated attack. The so-called propagation period is attained after 300 hours for DS 200 (Hf), after 550 hours for alloy 454 and around 620 hours for the MXON alloy. The alloy ranking thus established was confirmed by a detailed examination of the corrosion scale developed on each alloy. The specimens were examined at different test durations by optical and scanning electron microscopy ; the corrosion products were identified by X-ray diffraction. The corrosion attack follows a similar pattern for the three alloys :

a) formation of an external oxide layer associated with the development of a nickel-rich γ phase zone in contact with the alloy.

b) growth and subsequent local fracture of the oxide layer and internal sulphidation of the γ phase.

c) development of a very thick corrosion scale with a porous external oxide layer, a mixed zone containing a mixture of metal and oxides and a γ phase layer containing sulphides.

These three stages were observed in all the three alloys but at different test durations. It is important to note that in DS 200 and alloy 454 internal sulphidation starts after about 200 hours resulting in an
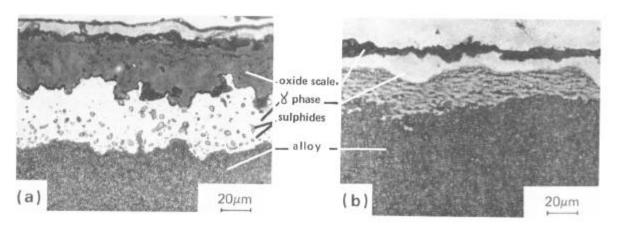
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Fig. 10 (a) and (b) — Corrosion scale morphology after 600 hours at 850°C. (a) alloy 454 ; (b) MXON.

accelerated attack of DS 200 whereas the corrosion rate of alloy 454 remains moderate until 600 hours. In the MXON alloy the γ phase is free of internal sulphidation even after 600 hours. The corrosion scale morphologies for the two single crystal alloys are compared in Fig. 10 after 600 hours. These results show that the external oxide layer formed on the MXON alloy is a more efficient barrier between the alloy and the salt than the scale formed on alloy 454. On both the alloys the main corrosion products detected after about 100 hours were Al_2O_3, $Ni\ Al_2O_4$, NiO and $Na\ Ta\ O_3$. However, on alloy 454, Al_2O_3 is no longer detected after 200 hours whereas this protective oxide is still present after 600 hours on MXON. It is believed that the greater stability of Al_2O_3 on the latter alloy is due to its initial higher aluminium content.

Conclusions

The present investigation has shown that it is possible to develop a titanium-free single crystal superalloy whose creep strength is substantially higher than that of the currently available conventional single crystals. More importantly, this work points out at the high creep potential of some alloy compositions containing appreciable amounts of chromium and aluminium. A further investigation of the role of cobalt shows that the elimination of this element improves the overall creep behaviour in the temperature range 760-1050°C. At 760°C, the alloy containing 7.5% cobalt shows a dramatic increase in the extent of primary creep compared to the alloys containing either 5% cobalt or none. The anomalous behaviour of MXON-7.5 Co alloy arises from the extended inhomogeneous {111} <112> slip during the very early stages of primary creep. Prior chemical homogeneity of the material affects the subsequent precipitation of a small amount of tungsten-rich μ phase at various temperatures. The tendency for μ phase precipitation is the greatest in the MXON-7.5Co alloy. Correspondingly, this alloy has the worst creep resistance. However, the precipitation of this phase can be avoided by using longer homogenizing durations. Interestingly, the presence of μ phase does not influence the high cycle fatigue behaviour of the MXON alloy since the crack initiation is governed by the micropores. This alloy shows excellent cyclic corrosion resistance at 850°C due to the development of a stable oxide layer which retards internal sulphidation. The cobalt-free version should provide the basis for further investigation.

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