EFFECT OF µ PHASE ON THE MECHANICAL PROPERTIES

OF A NICKEL-BASE SINGLE CRYSTAL SUPERALLOY

M. Pessah, P. Caron and T. Khan

Office National d'Études et de Recherches Aérospatiales (ONERA)

BP 72 - 92322 Châtillon Cedex - France

Abstract

The effect of the precipitation of μ phase particles on the mechanical properties of <001> single crystals of high strength MC2 superalloy was investigated. Plate-like and globular particles of a rhombohedral, W-rich, topologically close-packed (TCP) μ phase precipitate within the dendrites during exposure in the temperature range 800-1100°C, together with cuboidal M_6 C carbides in a few interdendritic subgrain boundaries. The presence of μ phase does not affect the fracture mechanism during the stress-rupture tests. Some tensile and high cycle fatigue tests were carried out on specimens previously exposed during 200 hours at 1050°C at a stress of 80 MPa and the results are compared to those obtained on unaged material. The decrease in yield strength observed at room temperature and 650°C is attributed to the coalescence of the strengthening γ ' phase rather than to a softening effect due to the precipitation of μ phase. Neither the tensile ductility nor the high cycle strength are affected by the presence of the TCP phase. A 200-hour exposure at 1050°C increases the impact strength at room temperature and 750°C of MC2 single crystals, due to the coarsening of the γ ' phase.

Introduction

The high level of the stress-rupture properties of recently developed nickel-base single crystal superalloys is mainly due to their large contents of refractory elements such as Mo, Re and W (1-4). However, excessive alloying additions may render the alloys prone to the formation of topologically close-packed (TCP) phases, such as μ , σ or Laves phases (4-6). In many cases, these TCP phases were shown to have deleterious effects on tensile, impact and stress-rupture properties of polycrystalline superalloys (5). These undesirable effects are : i) intergranular or transgranular cracking due to the brittle nature of the TCP phases ; ii) softening of the alloy because the TCP phases tie up some amounts of the refractory elements out of the γ matrix. Although alloy designers generally try to avoid the precipitation of TCP phases by a judicious control of the alloying elements, the very high-strength single crystal superalloys are expected to contain some amount of σ or μ particles. Since the information concerning the effects of TCP phases on the properties of single crystal superalloys is scarce in the literature, it was considered useful to undertake an investigation concerning the formation of μ phase particles in the MC2 alloy developed at ONERA and to determine how they subsequently affect its mechanical properties.

Experimental Procedure

The MC2 single crystal superalloy is designed for use as a high strength turbine blade material with an excellent creep resistance at temperatures above 1000°C. This objective was achieved by finding an adequate balance between alloying elements, without adding rhenium, and by keeping a reasonable density of 8.6 g.cm⁻³ (1). Its composition evolved from a previously developed alloy designated MXON (4). This MC2 alloy offers a creep temperature advantage of 40 to 50°C over the CMSX-2 single crystal alloy, developed by Cannon-Muskegon, at temperatures above 1000°C.

Element	Ni	Cr	Co	Mo	W	Al	Ti	Та
Nominal	Bal.	8	5	2	8	5	1.5	6
Heat ES2274 (Aubert & Duval)	Bal.	7.89	5.08	2.11	7.99	5.05	1.49	5.96

Table I. MC2 chemical composition (wt.%)

The master heat ES2274 used in this study was supplied by Aubert &Duval. The chemical analysis of this one-ton heat is compared in Table I to the nominal composition of the MC2 alloy. Single crystal bars were cast by the withdrawal process both at ONERA and at MICROFUSION (Howmet Group). Primary crystallographic orientations of the bars were all within 6° of a <001> orientation. All of the test material received standard solution and γ ' precipitation heat treatments, i.e.: 1300°C/3 hours/A.C. + 1100°C/4 hours/A.C. + 850°C/24 hours/A.C. In the fully heat-treated state, the γ/γ eutectic pools are completely dissolved and the material contains a homogeneous distribution of cuboidal γ ' precipitates with a mean size of 0.4 μ m and a volume fraction of about 70% (1).

Microstructural observations were made by scanning electron microscopy (SEM) both in the secondary and back scattered electron modes, and by transmission electron microscopy (TEM) using JEOL 200 CX and JEOL 4000 FX microscopes operating at 200 and 400 KV, respectively. The μ phase particles and the carbides were analysed by using energy dispersive (EDS) and wavelength dispersive (WDS) spectroscopy of characteristic X-rays. Crystallographic analyses were conducted by using X-ray diffraction and selected area diffraction (SAD) in the transmission electron microscopes. Quantitative image analysis was used to determine the volume fraction of μ phase on SEM micrographs.

Tensile, high cycle fatigue (HCF) and stress rupture specimens were machined from single crystal bars cast at ONERA. Constant load tensile creep tests were performed in air on cylindrical specimens. The tensile tests were conducted in air at RT, 650 and 950°C on cylindrical specimens with an initial strain rate of $1.1.10^{-4}$ s⁻¹. Half of all the tensile specimens were first aged in air at 1050°C during 200 hours under a tensile stress of 80 MPa in order to develop a rafted γ/γ microstructure typical of creep conditions and to precipitate the μ phase. The high cycle fatigue tests were conducted under load control in air at 870°C and at a R ratio of 0.02 at 70 Hz on hour-glass specimens. Similarly, some HCF specimens

were first aged for 200 hours at 1050°C at a stress of 80 MPa in vacuum to avoid surface oxidation which may promote initiation of external cracks. Impact tests were conducted in air at room temperature and 750°C on Charpy V-notch specimens machined out of single cystal bars cast at MICROFUSION. The faces of the specimens were cut parallel to [001] crystallographic planes. Some bars were aged at 1050°C during 200 hours before machining, in the absence of an external stress because it was not possible to apply a unidirectional stress during aging of the Charpy specimens.

Experimental Results and Discussion

Creep Behaviour

Typical high temperature creep properties of fully heat treated [001] single crystals of the MC2 alloy are reported in Table II.

Temperature (°C)	Stress (MPa)	Time to 1% creep (hours)	Rupture time (hours)	
760	750	504	1972	
950	240	200	637	
1010	170	581	763	
1050	140	631	896	
1050	110	2210	2311	
1100	130	254	292	

Table II. Typical creep data of [001] MC2 single crystals.

Observation of stress-ruptured specimens by SEM in the back scattered electron mode showed that particles containing heavy elements precipitate both within the dendrites and along a few subgrain boundaries which separate groups of dendrites. Figure 1 shows the morphology of these precipitates in the specimen tested at 1050°C and 110 MPa during 2311 hours. Within the dendrites, the particles are either acicular, globular or of cuboidal shape (Figure 1a). Careful examination of the acicular-like particles on various sections of the specimens showed that they are platelets, essentially parallel to {111} planes of the matrix. The width of these particles is about 0.5 µm and their larger dimension may attain 25 µm. The size of the globular particles is close to 1 µm. Observation of several specimens showed that the re-

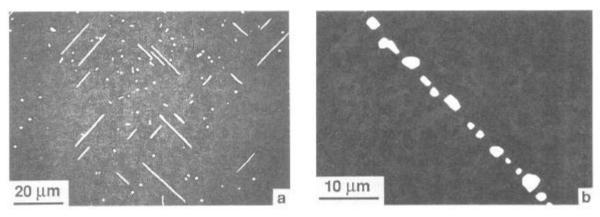


Figure 1 - SEM micrographs (back-scattered electron mode) showing particles rich in heavy elements in a MC2 single crystal after creep failure at 1050°C and 110 MPa for 2311 hours: a) within a dendrite (transverse section), b) along a subgrain boundary (transverse section).

Table III. Chemical analysis (wt.%) of μ phase, M₆C carbides, γ and γ ' phases in the MC2 alloy.

Element	Ni	Cr	Co	Mo	W	Al	Ti	Ta
γ	51.7	21.4	8.5	4.2	12	1.2	0.2	0.8
γ'	69	1.5	3.4	1	9.4	6.6	2.5	6.6
μ	16.9	9.5	4.2	11	53.5	0.1	0.5	4.3
M_6C^*	18.3	8.7	4.1	13.2	49.3	0.4	0.4	5.6

^{*} metallic elements

lative amounts of the three types of particles varied from specimen to specimen. In the subgrain boundaries, the particles are discrete and have a regular geometrical form with a size of about 1 µm (Figure 1b).

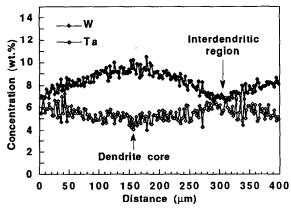
Electron diffraction, X-ray diffraction and X-ray microanalysis identified the plate-like and globular particles as μ phase precipitates and cuboidal ones as M_6C type carbides. The μ phase has a rhombohedral lattice with parameters close to a = 0.47 nm and c = 2.58 nm in a hexagonal description. The μ phase particles were found to have their hexagonal baseplane {0001} parallel to {111} or {120} planes of the matrix. The M_6C carbides have a fcc structure with a parameter close to 1.1 nm.

The results of the X-ray microanalyses of the metallic elements in the μ phase and in the M_6C carbides are shown in Table III. The μ phase is strongly enriched in W and contains Ni, Co, Mo and Cr. The chemical compositions of the γ and γ' phases in the MC2 alloy were analysed by atom probe time-of-flight mass spectroscopy (7) (Table III). The elements Co, Cr and Mo partition preferentially to the matrix, whereas W partitions nearly equally in the two phases. X-ray microanalysis across a dendrite on a tranverse section of a fully heat-treated [001] MC2 single crystal shows a residual segregation of W in the dendrite core, whereas the Ta content is higher in the interdendritic areas (Figure 2). The other metallic elements are distributed homogeneously in the dendrite. All these results illustrate that the μ phase particles precipitate from the γ matrix, preferentially in the region where the W level is maximum. This is confirmed by the fact that the μ phase particles are imbedded in γ' phase, the depletion of the matrix in elements Cr, Co and Mo leading to the transformation $\gamma \rightarrow \gamma' + \mu$.

The precipitation of μ phases was reported to occur in a number of γ or γ/γ' Ni and Fe-Ni based superalloys containing high levels of W and Mo (4-6, 8-14). A W-rich μ phase with a composition similar to that of the μ phase found in the MC2 alloy was identified by Yulin and Yunrong (12) in a series of conventionally cast nickel based superalloys containing various contents of W and Mo. These authors emphasize that the occurence of the μ phase does not depend only on the value of \bar{N}_{v} , average electron hole number for the matrix (5), but also on the values of both (Mo+W) at.% and Mo/(Mo+W) ratio. Taking the respective concentrations of W (3.2 at.%) and Mo (1.25 at.%) in the core of a dendrite of the fully heat-treated MC2 single crystal superalloy, (Mo+W) = 4.45 at.% and Mo/(Mo+W) = 0.28. These values agree with the results of Yulin and Yunrong who observe μ phase formation for critical values of (Mo+W) and (Mo/Mo+W) around 4.32-4.33 at.% and 0.27-0.28, respectively .

In the MC2 alloy, the concentration of metallic elements in the M_6C carbides is very close to the composition of μ phase. The analysis of the carbon content in several M_6C particles indicates values between 15 and 30 at.%. The presence of these carbides is in accordance with the fact that alloys which form M_6C are prone to the formation of μ phase (5). Indeed, there is a close structural relationship between the μ phase and the carbide M_6C ; for example, the coexistence of μ phase and M_6C carbides was reported in HASTELLOY® C-276 (13). Moreover, careful examination of W-rich particles in the dendrites shows that μ phase platelets are sometimes linked to carbide particles, which supports the hypothesis that μ phase can nucleate on M_6C carbides.

To determine the domain of occurrence of the μ phase in the MC2 alloy, fully heat-treated single crystal samples were held at temperatures between 750 and 1150°C for various times. The time temperature transformation (TTT) curve in Figure 3 shows that the formation kinetic of the μ phase is maximum in the temperature range 1000-1100°C and that μ phase was never observed after aging at 750 and 1150°C. At 1050°C, the volume fraction of μ phase attains a maximum value of 0.5 % after an exposure of 200



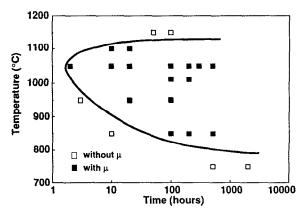


Figure 2 - Variations of W and Ta concentrations Figure 3 - TTT curve for the formation of μ phase across a dendrite of a fully heat-treated MC2 single crystal.

in the MC2 alloy.

hours and does not increase for longer periods of time. On the other hand, the volume fraction of μ phase measured in a sample exposed during 200 hours at 1050°C under a stress of 80 MPa is only 0.1% which shows that the stress may slow down the precipitation of μ phase in the MC2 alloy. Furthermore, the amount of μ phase observed in the specimen stress-ruptured after 896 hours at 1050°C is about 0.5%, i.e. similar to that measured after an exposure of 200 hours at the same temperature without stress.

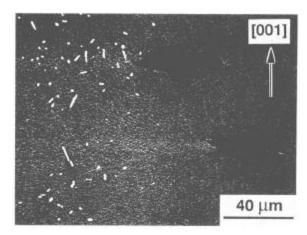
The effect of stress on the precipitation of TCP phases is still in debate. Some studies show that stress enhances σ phase precipitation in nickel based superalloys (5), whereas others reported an opposite effect (15). It appears that this effect must be complex and will depend on various factors such as temperature, alloy composition and nature of the TCP phase.

It is frequently suggested that the precipitation of TCP phases would reduce the stress-rupture strength of the superalloys by scavenging Mo, Cr and Co from the y matrix, thereby lowering the solution strengthening effects. It is generally considered that this softening effect is the major weakening mechanism in the case of μ phase precipitation (5). It is obvious that a significant softening effect will occur only if the amount of TCP phase exceeds a critical value. In the particular case of the MC2 alloy, this softening mechanism would not result in a strong decrease of the creep strength, because the amount of μ phase formed under stress is small.

In order to confirm this hypothesis, an attempt was made to prevent the formation of μ phase in a modified version of the MC2 alloy by decreasing the W content. It was found, however, that the μ phase could not be totally eliminated and that the stress-rupture strength of the alloy was reduced over the whole temperature range 760-1050°C. These results underline the difficulty of evaluating the softening effect caused by the precipitation of the TCP phases, since the compositional modifications influence the strength of the alloy, independent of the formation of the μ phase.

Another deleterious effect which could be expected due to the presence of μ phase platelets is the initiation of cracks at these particles, leading to a premature rupture of the alloy and thus to a reduction of the creep life. This weakening mechanism is generally associated with the σ phase, and it is considered to be dominant at low temperatures and high strain rates (5, 16). This mechanism is more specifically related to the grain boundary σ platelets which promote initiation and propagation of intergranular fracture which is not likely to occur in the MC2 single crystal alloy because the \(\mu \) phase platelets precipitate within the dendrite and only small discrete M₆C carbides form in a few interdendritic regions.

It is however worth mentioning that such an embrittling effect was reported during stress rupture tests of Ni-Cr-W-Mo austenitic alloys (8) where preferential nucleation of cracks was observed at interface between γ matrix and μ phase particles. Nevertheless, it must be stressed that the volume fraction of μ phase in these alloys was high (5 to 7 vol.%). Figure 4 illustrates the microstructure close to the fracture surface of a MC2 single crystal specimen crept at 1050°C and 140 MPa. Stress-rupture failures initiate in the interdendritic areas which are free of μ phase, and propagate normal to the stress axis, as in the



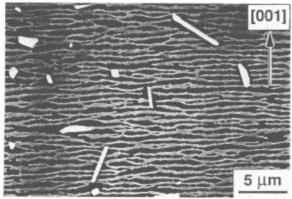


Figure 4 - SEM micrograph of the longitudinal section of a stress-ruptured MC2 single crystal specimen (T = 1050° C, $\sigma = 140$ MPa, $t_{\Gamma} = 896$ hours).

Figure 5 - Longitudinal section of a MC2 [001] single crystal exposed during 200 hours at 1050°C at a stress of 80 MPa.

TCP-free single crystal material. Furthermore, a detailed analysis of the platelike μ particles showed that no cracking occured along the phase boundary, although a few platelets were found to be broken, even far away from the rupture zone, illustrating the intrinsic brittleness of the μ phase. These observations suggest that the presence of μ phase platelets has no effect on the creep rupture mechanism of the MC2 alloy, inspite of their intrinsic brittleness.

Tensile Behaviour

Table IV shows the effect of a 200-hour exposure at 1050°C at a stress of 80 MPa on the tensile properties of fully heat treated MC2 [001] single crystal specimens. This aging heat treatment was applied to simulate engine service conditions in the airfoil. The microstructure of a stress-aged MC2 single crystal is shown in Figure 5. The γ ' precipitates have coalesced as rafts normal to the stress axis and μ phase particles are formed within the dendrites. The morphology and the distribution of the μ phase particles and M_6C carbides are comparable to those observed in samples aged without stress. The volume fraction of μ phase was evaluated to be about 0.1%.

Typical engineering stress/strain tensile curves are reproduced in Figure 6. The effect of this prior exposure is to decrease the yield strength only at RT and 650°C, whereas the UTS is significantly reduced at 950°C. On the other hand, the ductility is higher both at RT and 650°C for the aged specimens, whereas it is slightly decreased at 950°C.

Table IV. Effect of a 200-hour exposure at 1050°C at a stress of 80 MPa on the tensile properties of [001] MC2 single crystals.

	В	efore expos	ure	After exposure			
Temperature (°C)	0.2% Y.S. (MPa)	U.T.S. (MPa)	Elongation (%)	0.2% Y.S. (MPa)	U.T.S. (MPa)	Elongation (%)	
25	1 146	1 292	9.0	887	1 213	13.1	
25	1 133	1 214	9.1	854	1 286	14.5	
650	1 089	1 299	6.1	967	1 171	10.9	
650	1 070	1 261	6.1	998	1 228	9.5	
950	616	793	15.2	656	662	13.1	
950	659	799	16.7	621	665	10.1	

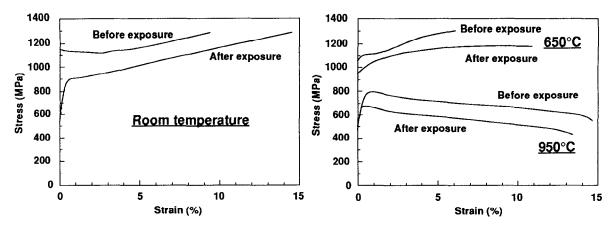


Figure 6 - Effect of a 200-hour exposure at 1050°C at a stress of 80 MPa on the tensile curves of MC 2 single crystals.

The decrease in yield stress for the aged material could be associated with the coalescence of the γ ' precipitates and/or with the precipitation of the μ phase particles. A significant softening effect due to the formation of a small amount of μ phase is excluded, as in the case of the stress-rupture strength. On the other hand, the coarsening of the γ ' phase tends to reduce the yield stress, because it promotes the Orowan by-passing mechanism of the precipitates by a/2<110> matrix dislocations instead of the cutting mechanism which is predominant in single crystal superalloys containing finely dispersed γ ' particles (16, 17).

This hypothesis is supported by the fact that the plateau region following the yield drop observed at room temperature and 650°C on the tensile curves of the unaged MC2 specimens is supressed after exposure. Indeed, these tensile instabilities previously observed in some single crystal superalloys (17, 18) were related, in the case of the CMSX-2 alloy, to the heterogeneous character of deformation concentrated in localized bands where the γ ' precipitates are sheared by pairs of a/2<110> dislocations bound by high energy antiphase boundary faults (17). The stage of work hardening occurs only when the deformation has spread through the entire specimen. The suppression of the yield drop and the plateau region on the tensile curves of the aged specimens is thought to be due to the homogeneity of deformation associated with the transition from the cutting mechanism of precipitates to the Orowan bowing deformation mechanism by dislocations gliding in the matrix between the γ ' rafts. Moreover, the increase of the tensile ductility suggests that the deformation is more hemogeneous in the aged material.

At 950°C, the yield stress of the MC2 single crystal alloy is not affected by prior exposure, but the ultimate tensile strength is significantly reduced compared with that of the unaged material. At this temperature bypassing of the γ ' precipitates by matrix dislocations operates primarily by a combination of slip and climb mechanisms. Interactions between the different operating slip systems lead to the formation of dense dislocation networks surrounding the γ ' precipitates (19), subsequently resulting in a strong work hardening, especially in the case of the alloy containing a fine dispersion of cuboidal precipitates where the total area of γ/γ ' interfaces is very high. In the case of the aged specimens, the work hardening is significantly reduced, presumably due to the smaller area of γ/γ ' interfaces in the rafted microstructure.

The precipitation of TCP phases was often reported to reduce the tensile ductility of superalloys by the embrittling mechanism described previously. Such a behaviour was observed in the case of the IN 100 alloy containing various amount of σ phase (15). The present investigation shows that the formation of a small amount of μ phase particles does not affect the tensile ductility of MC2 single crystal superalloy. Analysis of fracture surfaces showed that the rupture mode is not affected by the prior exposure at 1050° C, although μ phase particles appearing on the fracture surface are broken in many segments (Figure 7a). Observation of the microstructure of a tensile specimen broken at room temperature also showed a number of μ phase segmented platelets (Figure 7b). It is important to note that the cracks neither propagate in the matrix nor along the interphase boundaries.

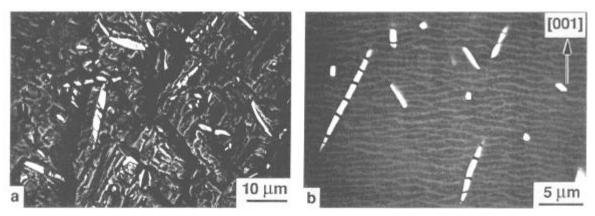


Figure 7 - MC2 tensile specimen tested at room temperature after a 200-hour exposure at 1050°C at a stress of 80 MPa; a) fracture surface, b) microstructure (longitudinal section).

High Cycle Fatigue Behaviour

The high cycle fatigue behaviour of MC2 [001] single crystals exposed during 200 hours at 1050° C at a stress of 80 MPa was evaluated in order to determine wether the presence of μ phase platelets initiates cracks leading to a premature failure of the material. Comparative tests were also undertaken on unaged specimens. The results of the tests conducted at 870° C with a maximum stress of 700 MPa are reported in Table V. HCF life is not affected by prior exposure at 1050° C under stress. Fracture analyses showed that the cracks initiate invariably at internal interdendritic casting porosity and never on μ phase particles. The micrograph of Figure 8 illustrates the fracture surface of an aged specimen. The crack propagates in Stage II before the final rupture occurs in Stage I along $\{111\}$ cristallographic planes.

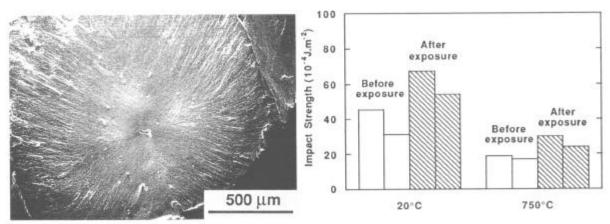
Impact Tests

The effect of a prior exposure at 1050°C during 200 hours on the impact strength of MC2 [001] single crystals was investigated both at room temperature and 750°C. The results of these tests are shown in Figure 9. The aging treatment causes an improvement of the impact strength of the material, both at room temperature and at 750°C. As in the case of tensile tests, the homogeneity of the deformation in the coarsened microstructure is very likely responsible for this improvement in toughness. Analysis of the fracture surfaces showed no differences between the aged and unaged single crystals, except for the presence of broken μ phase particles in the aged material as previously observed in tensile specimens. The small decrease of the impact resistance between room temperature and 750°C agrees with the results reported on RR 2000 and SRR 99 single crystal superalloys (20). It has also been reported that the impact properties of the CMSX-2 single crystal alloy were significantly improved by pseudo engine service temperature exposure (21).

These findings on single crystal superalloys do not corroborate the results reported by Yulin and Yunrong (12) on cast nickel based superalloys. These authors indeed observed a reduction of impact strength after exposure at high temperature of alloys prone to μ phase formation. Similarly, the impact toughness of iron based alloys was reported to be strongly reduced in the presence of μ phase (9). In this

Table V - Effect of a 200-hour exposure at 1050°C at a stress of 80 MPa on the high cycle fatigue life at 870°C of MC2 [001] single crystals (maximum stress = 700 MPa, R = 0.02, f = 70 Hz).

Number of Cycles to Failure			
Before exposure	After exposure		
168 000	327 000		
394 056	343 000		



at 870°C (maximum stress = 700 MPa, R = 0.02, ture and 750°C of [001] MC2 single crystals. f = 70 Hz).

Figure 8 - High cycle fatigue fractograph of a Figure 9 - Effect of a 200-hour exposure at MC2 single crystal specimen aged before testing 1050°C on the impact strength at room tempera-

case, however, fracture analysis showed that the precipitation of a small amount of μ particles (0.1 to 1wt.%) in the grain boundaries caused an embrittlement effect by promoting intergranular fracture.

Conclusions

The present investigation has shown that it is possible to tolerate a small amount of μ phase particles in the MC2 single crystal superalloy without affecting its mechanical properties although the upper tolerable limit of this phase is yet to be determined. The salient features emerging from this study are:

- 1. Plate-like and globular particles of a rhombohedral, W-rich, μ phase precipitate within the dendrites of the MC2 superalloy during exposure in the temperature range 800-1100°C. The volume fraction of μ phase is 0.5% after a 200-hour exposure at 1050°C without stress. Moreover, cuboidal M6C carbides precipitate along some interdendritic subgrain boundaries.
- 2. Application of a tensile stress along a <001> orientation during exposure at high temperature decreases the amount of μ phase and promotes oriented coarsening of the γ phase as rafts normal to the stress axis.
- 3. The µ phase particles are brittle, but this does not affect the rupture mode and the ductility of the MC2 single crystals during tensile, stress-rupture and impact tests. The µ phase platelets do not initiate failure during HCF tests at 870°C.
- Oriented coarsening of the γ' phase decreases the yield strength of the MC2 single crystal alloy at room temperature and 650°C and increases its impact strength at room temperature and 750°C.

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