K. Firlus et al.: The influence of Cr, Al, Co, Fe and C on negative creep of Waspaloy

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The influence of Cr, Al, Co, Fe and C on negative creep of Waspaloy

Negative creep using precision dilatation experiments is investigated on a broad variety of single, dual and multi-phase nickel-based superalloys. Pure nickel and dilute binary nickel-based alloys show no signs of negative creep. However, with higher contents of Cr and Al and in highly alloyed ternary and multicomponent nickel-based alloys negative creep is observed. Short range ordering of Cr and/or Al are identified to cause negative creep at 550 °C. Carbon additions leading to retarded carbide precipitation or transformation can enhance negative creep.

Keywords: Nickel-based superalloy; Negative creep; Carbide transition; Short range ordering

1. Introduction

The phenomenon of volume contraction during isothermal annealing without mechanical load was first described by Sykes [1] on binary FeW-alloys in 1926. Later, Fountain and Korchynsky [2] introduced the term negative creep for this phenomenon. They investigated the creep behaviour of a solution annealed Fe-alloy with 20.5 at.% W and a Ni-alloy with 25.5 at.% Mo at 600 °C. Although the specimens were under tensile loading, a negative strain was measured. This contraction is explained by the formation of the phases Fe₂W and Ni₄Mo. Later, extensive investigations of nega-

tive creep on the nickel-based alloys Ni-20 % Cr and Nimonic 80A were conducted [3–6]. Following that, negative creep was investigated on many nickel-based alloys using stress free, isothermal and discontinuous annealing [7–14].

Thomas [21] observed anomalies in the temperature dependence of the electrical resistance in NiAl, NiCr and some other binary solid solution alloys where the solvent is a transition metal such as Ni. He observed that the electrical resistivity increases during ageing heat treatments at intermediate temperatures compared to that in the solution-treated condition. The formation of a distinct structural state in the solid solution called the “complex phase state” or “K-state” was seen as the cause for this increase in resistivity. Later the K-state was attributed amongst others to heterogeneous short-range-ordering which is often observed in alloy systems at the boundaries of known order–disorder transitions [22]. Guy [23] found with diffuse X-ray scattering that SRO is also involved in the formation of the K-state in some NiAl-alloys. Starke et al. [24] observed the formation of a K-state, i.e. an increase in the electrical resistance, of a solid solution of Ni with 8.2 at.% Al during ageing at 400 °C. They too attributed the K-state to SRO.

Timmins et al. [14] investigated the creep behaviour of Nimonic 90 at 850 °C and measured a negative strain of 0.05 % after 800 h. The authors exclude order / disorder phenomena because they are supposed to occur exclusively below 600 °C. Instead, changes in the γ' -volume fractions are presented as the cause of negative creep.

In his work on IN 738, Reppich [10] states that there is no connection between negative creep and changes of either the morphology or the volume fraction of the γ' -phase. Instead, minor phases present in IN 738 provoke negative creep. Reppich [10] as well as Kinzel et al. [13] emphasise the role of the transformation of MC type carbides to $M_{23}C_6$ type carbides after the following equation:

where γ and γ^* are both face centred cubic (fcc) matrix phases but with different compositions. In the case of IN 738, the carbide transition leads to an elongation [10], whereas a contraction is observed for Waspaloy [13].

The causes of negative creep in nickel-based alloys given in literature can be summed up with ordering effects in Cr and/or Al containing Ni-based alloys [5, 15, 16, 25], evolution of the γ' volume fraction in Al and/or Ti containing Ni-based superalloys [12, 13] and carbide transition from MC to $M_{23}C_6$ [10, 12, 13].

However, consistent reasoning for negative creep in nickel-based alloys is still missing. Instead, given explanations often seem contradictory. Therefore, the goal of this study is to get a better understanding of negative creep in nickel-based alloys. In order to test various hypotheses for the causes of negative creep, pure nickel and a variety of dilute nickel alloys were investigated on the one hand. On the other hand, highly alloyed complex nickel-based alloys derived from the superalloy Waspaloy were investigated.

2. Experimental

The dilute nickel-based alloys listed in Table 1 and the complex nickel-based alloys MA_p and MA* (Table 2) were produced by melting pure elements (min. 99.9%) in an Ar-arc furnace. To get homogeneous distributions of the elements, the alloy buttons were flipped and re-melted five times. Subsequently, the dilute nickel-based alloys were solution annealed at 1200 °C for 8 h in air atmosphere, followed by quenching in oil. Specimens for scanning electron microscopy (SEM),

Table 1. Composition of pure nickel and dilute nickel-based alloys. All alloys in this table are cast polycrystalline.

Label	Element in at. %					
	Ni	Al	Ti	Co	Mo	Cr
Ni (Reference)	100	–	–	–	–	–
NiAl5	95	5	–	–	–	–
NiAl15	85	15	–	–	–	–
NiCr5	95	–	–	–	–	5
NiCr10	90	–	–	–	–	10
NiAlTi	94.6	2.2	3.2	–	–	–
NiAlTiCo	82.2	2.2	3.2	12.4	–	–
NiAlTiCoMo	78.3	2.2	3.2	12.4	3.9	–

Table 2. Composition of Waspaloy, master alloy and modified master alloys. All alloys in this table are cast SX except MA_p and MA*. MA = master alloy, MA_p = master alloy polycrystalline and MA* = master alloy without Cr in polycrystalline state.

Label	Element in at. %							
	Ni	Cr	Co	Mo	Al	Ti	Fe	C
Waspaloy	49.8–60.6	19.7–22.9	11.6–14.4	2.1–3.0	2.5–3.4	3.3–4.5	0–2	0.02–0.1
MA, MA _p	56.4	19.5	12.1	2.3	4.7	3.9	1.0	< 0.011
MA*	75.3	0.0	12.4	2.4	4.8	3.9	1.1	< 0.011
C0.9	55.8	19.3	12.0	2.3	4.6	3.8	1.0	0.9
C2.3	55.1	19.0	11.8	2.3	4.6	3.8	1.0	2.3
Cr24	53.0	24.2	11.3	2.2	4.4	3.7	1.0	< 0.011
Cr29	49.9	28.5	10.8	2.1	4.2	3.5	0.9	< 0.011
Co15	54.7	18.9	14.6	2.3	4.5	3.8	1.0	< 0.011
Co18	52.9	18.3	17.6	2.2	4.5	3.6	0.9	< 0.011
Fe3	55.5	19.1	11.9	2.3	4.7	3.8	2.6	< 0.011
Fe4	54.7	18.9	11.7	2.2	4.5	3.7	4.1	< 0.011
Mo4	55.6	19.2	11.9	3.6	4.7	3.9	1.0	< 0.011
Mo5	55.0	18.9	11.8	4.9	4.6	3.8	0.9	< 0.011

X-ray diffraction (XRD) and dilatometry were cut out via wire eroding. Continuous dilatation experiments were done in a dilatometer DIL 402C from Netzsch at 550 °C, a temperature at which negative creep has often been observed in literature [12, 13, 20, 25–27] to occur in nickel-based alloys.

A large ingot of the so-called master alloy (MA) was produced via vacuum induction melting (VIM) to attain highest purity. The master alloy composition falls within the specified composition range of Waspaloy although the contents of the major alloying elements are generally at the lower limits (Table 2). The carbon content of < 0.011 %, in particular, is at the lowest technically possible level by the VIM production technique and was measured at IME in Aachen according to DIN EN ISO 15350 using an infrared absorption method after combustion in an induction furnace.

The master alloy ingot was divided and sections were subsequently remelted with various C, Mo, Cr, Co and Fe additions in an Ar-arc furnace. Finally, these alloys batches were recast under vacuum into single crystalline (SX) rods with a diameter of 16 mm and a length of 150 mm using the Bridgman process with the setup from [28]. The parameter for the single crystal casting were: 0.01 mbar, 1450 °C and a withdrawal rate of 3 mm · min⁻¹. The compositions and denominations of these SX-alloys are given in Table 2.

The single crystals and the polycrystalline alloys MA_p (p for polycrystalline) and MA* (master alloy with 0 at.% Cr) were subjected to the usual heat treatment for Waspaloy, i. e., a solution annealing at 1025 °C for 4 h with a subsequent quench in oil, followed by an anneal at 845 °C for 4 h and air cooling and annealing at 760 °C for 16 h and air cooled. After heat treatment, discs were cut from each SX-rod. The rest of these SX-rods were precisely turned down to rods with a diameter of 10 mm and a length of 100 mm ± 0.12 mm at MTU Aero Engines AG in Munich. High precision length measurements for these smaller rods were also done at MTU Aero Engines AG in Munich using a laser-measurement system (Mahr ULM Rubin 1500). To avoid thermal dilatation, the length measurements were carried out in an air-conditioned room. Rods and discs were sealed in a fused silica tube under vacuum to avoid oxidation as much as possible. Afterwards they were discontinuously annealed for 10, 100, 300 and 1000 h at 550 °C and subsequently air-cooled to room temperature.

To reveal the microstructures, specimens were ground, polished and etched with Mo-acid. Microstructure analysis was done with SEM (scanning electron microscopy) (Zeiss 1540 EsB) using secondary electron and backscattered electron detectors. The chemical compositions were investigated with energy dispersive X-ray spectroscopy (EDS). Carbides and the size of γ'-precipitates, were determined by stereological image analysis with the open source software ImageJ [29]. Thermo-Calc and the Database TTNi8 were used to predict the stable phases and their composition at given temperatures.

Crystallographic analysis was done via X-ray diffraction (XRD) with a Discover D8 Advance from Bruker using Cu-K_α radiation. The electrical resistance measurement took place in a climatic chamber at -40 °C with a linear bat-

tery testing for high current cell applications (LBT 5 V-25A from Arbit) using the 4-point probes method. Each specimen was first measured in the heat treated state to get the initial resistance ρ_0 and after ageing at 550 °C for 40 h ρ .

3. Results

Figure 1 shows the results of the dilatation experiments of pure Ni, the dilute nickel-based alloys and the polycrystalline alloys MA_p and MA* derived from Waspaloy. Within the measurement error Ni and NiCr5 show no contraction whereas the binary alloy NiAl5 reaches a steady contraction of $-0.5 \cdot 10^{-4}$ after about 10 h. Increasing the Cr content to 10 at.% shifts the dilatation to negative values. A higher Al content of 15 at.% on the other hand leads to an elongation compared to NiAl5.

Addition of Ti to NiAl alloy alters the behaviour significantly. For NiAl5 the maximum contraction was $-0.5 \cdot 10^{-4}$

Fig. 1. Relative length changes measured continuously with dilatometer at 550 °C. MA_p has the same composition as the master alloy but is cast polycrystalline. MA* is also polycrystalline but Cr is substituted with Ni.

Fig. 2. Relative length change of the master alloy and its derivatives at 550 °C.

but for NiAlTi with 2.2 at.% Al and 3.3 at.% Ti the maximum contraction is $-1.5 \cdot 10^{-4}$ after 40 h. Adding Co to the NiAlTi alloy does not affect the contraction. The same can be observed for NiAlTiCoMo.

The length changes of MA^* and MA_P are shown in Fig. 1, too. Both alloys undergo almost the same contraction of around $-1.1 \cdot 10^{-4}$ after 40 h at 550 °C.

The results of length measurement after a discontinuous annealing at 550 °C are shown in Fig. 2. All alloys show negative creep with the same curve progression. The rate of negative creep is highest within the first few hours. Even high content of 29 at.% Cr has no significant impact on the measured contraction.

In XRD patterns of the alloys Ni, NiAl5, NiCr5, NiCr10 and NiAlTi, only peaks of a fcc phase are found (see for example Fig. 3 for NiAlTi). Also, no hints for second phases are observed in these alloys. NiAlTiCo and NiAlTiCoMo show cuboidal γ' particles with around 50 nm size (Fig. 4).

The lattice parameters of the single-phase alloys showing negative creep are given in Table 3. After the heat treatment of 40 h at 550 °C the lattice parameter of NiAl5, NiAlTi and NiCr10 decreases, whereas for Ni and NiCr5 it is constant considering the measurement error.

Fig. 3. Diffraction Pattern of NiAlTi after 40 h at 550 °C.

In none of the investigated alloys derived from Waspaloy (alloys given in Table 2) was the 3step heat treatment sufficient to homogenize the dendritic cast structure (Fig. 5). In the primary dendrite arms, bimodal γ' -size distributions are observed (Fig. 5a). The bigger precipitates have a mean diameter of ~ 120 nm and the small precipitates have a mean diameter of ~ 40 nm. The dendrite cores generally show high contents of small γ' -precipitates with diameter of ~ 40 nm (Fig. 5b). Large γ' -precipitates with diameters > 200 nm are observed only in interdendritic zones (Fig. 5c). After 1000 h at 550 °C the microstructure remains unchanged.

With increasing C-content, the amount of Ti-carbides rises. These Ti-carbides are located only in the interdendritic areas. C0.9 and C2.3 also have an area in the core of the dendrite, in which only small γ' -precipitates are located. This region is many times smaller compared to the other alloys. Besides γ , γ' and (Ti,Mo)C, a fourth phase in the specimen C2.3 has formed (Fig. 6b). This phase only occurs in the interdendritic regions. It forms a network which resembles a “chinese script” morphology. An EDS investigation of the additional phase, as seen in Fig. 7, reveals that Ni is depleted and Cr, C and Mo are enriched. Furthermore, this mapping shows that the Ti and Mo concentration is high in the larger carbides, which is also the case for the C0.9 specimen.

Fig. 4. SEM image of NiAlTiCoMo after 40 h at 550 °C etched with Mo-acid.

Table 3. Cubic lattice parameters of the single-phase alloys. The measurement error is ± 0.1 pm.

Specimen	Lattice parameter (pm)		$\frac{d_{\text{annealed}} - d_0}{d_0}$ in 10^{-4}
	Initial state d_0	40 h at 550 °C d_{annealed}	
Ni	352.5	352.5	0
NiAl5	353.4	353.3	-2.8
NiAlTi	355.3	355.2	-2.8
NiCr5	353.4	353.4	0
NiCr10	353.7	353.5	-5.7

Fig. 5. (a) Overview and (b-d) zoomed in SEM pictures of the microstructure of MA and its derivatives after the 3-step heat treatment mentioned in materials and procedures. Primary (b) dendrite arm, (c) core of a dendrite, and (d) interdendritic area.

The (Ti,Mo)C volume fraction of C2.3 drops from $5.4 \pm 0.7\%$ after the heat treatment to $4.0 \pm 0.9\%$ after 1000 h at 550°C . For C0.9, the (Ti,Mo)C volume fraction is lower at a value of $1.0 \pm 0.5\%$ and stays constant even after an annealing for 1000 h.

Table 4 shows the results of the electrical resistance measurement. The resistance increased in NiAl5, NiCr10 and NiAlTi after ageing at 550°C for 40 h.

4. Discussion

4.1. Ni-Cr ordering

Pure Ni and single-phase NiCr5 show no negative creep whereas, a Cr-content of 10 at.% in NiCr10 leads to a small contraction after long time annealing. This contraction is caused by a decrease in the lattice constants as seen in Table 3, accompanied by an increased electrical resistance

which is a good indication for SRO [17–19, 30, 31]. In literature, the evolution of SRO with time is often held liable for the measured decrease of the lattice parameter in NiCr alloys [18]. It is also mentioned that the ordering kinetics slow down for alloys with a Ni/Cr-ratio deviating from 2/1 of the prototype phase Ni_2Cr [18, 19]. This explains why NiCr5 shows no sign of contraction whereas NiCr10 does.

The evolution of SRO is also often accounted for negative creep in Cr-containing nickel-based superalloys [6, 17, 25]. However, the dilatation curves are very similar to the alloys MA_p and MA^* (Fig. 1), both derived from Waspaloy, but MA_p containing 19.5 at.% Cr and MA^* containing no Cr at all. Hence, SRO of Ni_2Cr cannot be the sole cause of negative creep in Cr-containing nickel-based superalloys.

For times up to 1000 h, the Cr content does not influence the negative creep behaviour, as seen in Fig. 2. The master alloy MA has a Cr content of 19.5 at.% and Cr29 28.5 at.%. Considering that Mo, Cr, and W have a stabilizing effect for

Fig. 6. SEM pictures of C2.3 etched with Mo-acid. (a) Carbide structure, (b) TiC and the fourth phase in C2.3, and (c) zoomed in on the fourth phase.

Fig. 7. EDS of C2.3 after 100 h at 550 °C.

Table 4. Electrical resistance change after the heat treatment (ρ_0) and after ageing at 550 °C for 40 h (ρ).

	electrical resistance changes in $10^{-5} \Omega\text{m}$		
	NiAl5	NiCr10	NiAlTi
ρ_0	15.7	47.6	33.5
ρ	15.9	48.1	34.9
$(\rho - \rho_0)/\rho_0$ in 10^{-4}	127	105	418

forming the $\text{Ni}_2(\text{Cr}, \text{Mo}, \text{W})$ phase, even a combined amount of Cr and Mo in Cr29 of 30.6 at.%, which is reasonably close to the ideal ratio of 2 : 1 NiCr_2 , does not affect negative creep. This might be explained by the high amount of Co in these alloys, which has a destabilizing effect on the Ni_2Cr superstructure [32]. This would shift the SRO to LRO transition to longer annealing times.

4.2. Carbide transition

Reppich [10], as well as Kinzel et al. [12], suggested that negative creep may not be caused by any kind of time dependent alterations of the major phases but rather by alterations of minor phases, i.e. a carbide transition from MC to $M_{23}C_6$. To clarify these hypotheses, Waspaloy derivatives with var-

ious C contents were investigated. The C content is varied from less than 0.011 % in alloy MA up to 2.3 at.% C in C2.3. Figure 2 shows almost identical behaviour of relative length change for MA and C0.9. Only C2.3 with a very high C content shows a slightly stronger contraction up to 300 h. Besides (Ti,Mo)C carbides, EDX measurements strongly indicate that the fourth phase in C2.3 is a (Cr,Mo)₂₃C₆ carbide. This carbide forms relative quickly in C2.3 and can be seen after 100 h at 550 °C. Although C0.9 has a high volume fraction of carbides for a nickel-based superalloy, no (Cr,Mo)₂₃C₆ is observed in this alloy after 1 000 h at 550 °C. Therefore, no carbide transition took place, which explains why the MA and C0.9 negative creep curves overlap. Thus the carbide transition from MC to M₂₃C₆ is an additional mechanism for negative creep, but it is not necessary for negative creep as seen in the C free MA specimen.

4.3. Grain boundary effects and γ' morphology

The SX-alloys derived from Waspaloy all show negative creep. Therefore, grain boundary effects such as coarsening are neither the cause nor necessary for negative creep. A comparison between SX master alloy MA investigated in this study and commercial, polycrystalline Waspaloy investigated by Kinzel et al. [13] shows that, except for the measurement at 300 h, the stress-less creep curves are close. Waspaloy has a microstructure consisting of γ , γ' , TiC and Cr₂₃C₆. The γ' size is between 30 and 40 nm with a volume fraction of 26 vol.% [13]. However, the γ' morphology of MA is rather different as can be seen in Fig. 5. MA has a dendritic structure with different γ' sizes and geometries in the dendritic cores and arms compared to the interdendritic areas. Although the morphology and size of γ' differs considerably compared to polycrystalline commercial Waspaloy investigated by Kinzel et al. [13], the negative creep curves are very similar. Thus, the γ' morphology has only a minor or no influence on negative creep.

4.4. Negative creep in NiAl5, NiAl15 and NiAlTi

As seen in Fig. 1, NiAl5 contracts whereas NiAl15 does not. NiAl5 is a single-phase alloy and NiAl15 a two phase alloy containing fcc, γ -matrix and L1₂ ordered γ' Ni₃Al. The reason for the contraction of NiAl5 is the same as for NiCr10: SRO. Sitaud and Dimitrov [31] observed the kinetics of local ordering in two binary NiAl alloys containing 6 and 10 at.% Al. The ordering kinetics were indirectly determined through the measurement of the electrical resistivity at 4.2 K. After normalisation treatment for 30 min at 1 000 °C followed by furnace cooling and isothermal annealings for 30 min at 400 – 450 °C the residual resistivity rises, indicating SRO [31]. SRO in NiCr-alloys is accompanied by a decrease in the lattice parameter. This is also the case for the NiAl5 specimen in which the lattice parameter also decreases. The higher decrease of the lattice parameter compared to the measured contraction can be explained by the heating and cooling phase of the dilatometer in which ordering and therefore a decrease in the lattice parameter also occurs. While length measurement

inside the dilatometer is measured after reaching 550 °C and ends after 40 h at this temperature, the XRD measurements were done before and after the dilatometer measurements and therefore include the heating and cooling phase of the dilatometer. The results of the electrical resistivity done in this work indicate that SRO in NiAl5 is indeed the cause for negative creep. Although NiAl5 and NiCr5 are both single phase alloys, only NiAl5 shows contraction within the 40 h at 550 °C. One reason for that is the slower ordering kinetics of NiCr5 compared to NiAl5. If the Ni/Cr-ratio deviates from 2/1 of the prototype phase Ni₂Cr, the ordering kinetics in NiCr-alloys slow down [18, 19]. The other reason for that might be the higher diffusion coefficient of Al in Ni ($1 \cdot 10^{-16} \text{ cm}^2 \cdot \text{s}^{-1}$ extrapolated from the work of Allison and Samelson [33]) compared to Cr in Ni ($3 \cdot 10^{-18} \text{ cm}^2 \cdot \text{s}^{-1}$) [34] at 550 °C.

For NiAl15, more precisely the γ matrix in NiAl15, negative creep might also occur, but the formation of γ' works against the contraction in the matrix. After the solution annealing at 1 200 °C followed by quenching in oil the alloy NiAl15 is far from equilibrium. Thermo-Calc predictions give rise to the expectation that the γ' volume fraction in the quenched alloy is smaller than the equilibrium volume fraction at the annealing temperature of 550 °C. In a binary, two phase alloy containing Ni and Al, the lattice parameter of γ' is larger than γ and therefore, the misfit in these alloys is positive [35, 36]. Thus increasing the volume fraction of γ' during annealing at 550 °C will result in a net increase in the lattice parameter of NiAl15 explaining the macroscopic dilatation of the NiAl15 sample. For nickel-based superalloys with a positive misfit, i.e. Waspaloy [12, 13, 37], the formation of γ' can therefore mitigate the measured contraction.

Compared to NiAl some Al is replaced by Ti in alloy NiAlTi (Table 1). Even though the overall content of solute elements is almost the same, NiAlTi contracts considerably stronger than NiAl during annealing at 550 °C (Fig. 1). Tarfa et al. [30] investigated ordering in nickel-based solid solution alloys containing 4 at.% Al and 2 at.% Ta, 4 at.% Al and 2 at.% Ti as well as 1 at.% Al and 3 at.% Ti respectively. After isothermal annealing tests of various durations, residual resistivity measurements were carried out. Tarfa et al. [30] observed an increase in the electrical resistivity in all investigated alloys caused by SRO. In addition, according to Tarfa et al. [30], the substitution of Al by Ti leads to “a more extended SRO-evolution on the time scale” and “an increase in the activation enthalpy of ordering”. In these investigations too, a greater increase in the resistivity was found for NiAlTi compared to NiAl (Table 4). A stronger short range ordering in NiAlTi compared to NiAl is responsible for the stronger contraction and therefore, a more pronounced negative creep. Further additions of Co and Mo to NiAlTi have no significant impact on negative creep.

5. Conclusions

In this study, negative creep in nickel-based alloys was investigated. Pure Ni, the dilute solid solution NiCr5 as well

as NiAl15 showed no contraction, whereas NiCr10, NiAl5 and higher additions derived from Waspaloy show negative creep. It turned out that short range ordering of Cr and Al additions are responsible for negative creep in all these alloys. A carbide transition from MC to $M_{23}C_6$ can enhance negative creep but is not the sole cause for negative creep in nickel-based alloys. In alloys with a positive misfit, an increasing γ' volume caused by a further approach to equilibrium in the course of an annealing, can counteract negative creep. Therefore, negative creep of the matrix in NiAl15 is masked by a volume fraction increase of γ' during isothermal annealing at 550 °C.

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