

Development of a new 718-type Ni-Co superalloy family for high temperature applications at 750°C

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Abstract. Alloy 718 has been used for many years in the aerospace industry due to its unique mechanical properties and good processing characteristics, especially its workability. However, the temperature limit of Alloy 718 is about 650 °C because of the thermal instability of the main strengthening phase γ'' -Ni₃(Nb,Ti,Al). Numerous attempts have been made to develop a new wrought 718-type alloy for high temperature applications. The approach was to increase the stability, i.e. the solvus temperature of the γ' -phase ($T_{\gamma',s}$). However, this affected workability as the solvus temperature of the δ -phase ($T_{\delta,s}$) did not increase accordingly so that the window for fine grain forging $T_{\delta,s}-T_{\gamma',s}$ became smaller. In this paper the development of a new γ'/γ'' -alloy on the basis of Alloy 718 is presented, where the microstructure is stable at 800 °C, mechanical properties are similar to Alloy 718, yet do not deteriorate beyond 650 °C, and the forging window is wider than the one of Alloy 718, allowing for good workability. This was essentially achieved by the addition of about 17%–30% Co in combination with an Al/Ti-ratio of more than 5.0 and an Al-content of about 1.6%–2.2%. The key role of cobalt is to stabilize the δ -phase, allowing for solvus temperatures in excess of 1100 °C. Consequently, the stability of the γ' -phase can be increased by further addition of aluminium. At the same time the Ti-content is reduced to prevent formation of the η -(Ni,Co)₃(Ti,Al,Nb) phase. Besides discussion of the alloy development concept, information on microstructure evolution and mechanical properties will be given.

1. Introduction

The past decade has seen increasingly rapid advances in the field of high temperature disk alloys for aircraft engines and stationary gas turbines [1,2].

The main aim of these advances is the development of a new alloy which can be used at temperatures of up to 725 °C and can be processed by normal industrial wrought processes. In most of these studies Alloy 718 has been chosen as the basis alloy because of its excellent combination of mechanical properties, good machinability – especially workability – and relatively low cost. The unique good workability of Alloy 718 is based on the existence of the δ -Ni₃Nb phase which makes fine grain forging (ASTM 8 and finer) possible. The precipitations of the δ phase pin the grain boundaries and avoid grain coarsening during forging; for this reason the last forging step of Alloy 718 is usually done at temperatures just below the delta-phase solvus in order to preserve the material's relatively low resistance to deforming and ensure a fine grain size at the same time [3]. The solvus temperatures of the γ'' and of the γ' phases are about 920 °C and about 850 °C, respectively. The γ'' phase is not relevant for the forging process of Alloy 718 because of the semi-coherency and the slow precipitation kinetic brought by it. The main relevant strengthening phase for the workability process is the γ'

phase. Due to coherent precipitation, hardening occurs very quickly and material strength grows rapidly. In this case the alloy needs to be tempered at a higher temperature in order to dissolve the precipitated γ' particles.

The temperature range in which fine grain forging is possible between the solvus temperature of the delta phase and the solvus temperature of the strengthening phase is called the forging window. In this range grain coarsening is still avoided but the material's resistance to deformation is low enough for forging. The forging window of Alloy 718 has a range of about 180 °C.

However, Alloy 718 can only be used at temperatures up to 650 °C due to its microstructural instability. The metastable γ'' phase overages rapidly and transforms to the thermodynamically stable δ phase at temperatures above 650 °C. In doing so the alloy loses its strength and ductility because of the formation of the plate-like δ phase particles in the grain.

The most established way of stabilizing the microstructure in the alloys is by increasing the amount of the γ' phase. For applications at temperatures of up to 700–750 °C, Waspaloy is usually used and Alloy 718Plus is a possible candidate under consideration. These two alloys have an increased amount of γ' phase which allows their use at higher temperatures than Alloy 718 [4,5]. In Alloy 718Plus the δ and η phases [6–8] can precipitate on the grain boundaries same as in Alloy 718 to avoid the grain coarsening.

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However, increasing the amount of the γ' phase increases its solvus temperature to about 950 °C in Alloy 718Plus [7]. This reduces the forging window to around 100 °C because the solvus temperature of the δ phase remains constant. Thus, the forging process becomes more difficult and expensive, because of raised numbers of tempering, in comparison to Alloy 718.

In Waspaloy the amount of the γ' phase is raised even further and thus the solvus temperature of the γ' is about 1030 °C. Furthermore, $M_{23}C_6$ carbides are used as the 2nd high temperature phase for pinning grain boundaries. However, the solvus temperature of $M_{23}C_6$ carbides is about 1030–1040 °C and consequently, Waspaloy does not really have a fine-grain forging window [9]. For this reason, it is not possible to achieve similarly fine grains as in Alloy 718. Furthermore, its processing costs are much higher than for Alloy 718, because the alloy needs a lot of heat treatment during forging.

The aim of this research is the development of a new design concept for a nickel-based wrought superalloy family for high temperature applications in aero engines and stationary gas turbines at a maximum temperature of 750 °C. The wrought properties of the new materials should be comparable to Alloy 718, so that fine grain forging is possible with ease. For this reason, the forging window must be larger than 100 °C.

The current work focuses on the development concept assisted by ThermoCalc simulations (Version7, Database TTNi7), supported by experimental work. The influence of Co, Al and Ti on the microstructure and the microstructural stability of alloys at temperatures of 750 °C and 800 °C for 500 h and 2000 h were studied.

2. Experimental procedures

In order to predict the phases and their solvus temperatures in the new alloys, ThermoCalc software was used. More than 50 different alloys were produced and used to experimentally study the effects of various elements on microstructure, forging- and mechanical properties, and microstructural stability at higher temperature. Most alloys presented in this paper were made through plasma arc melting, followed by casting the material into a water-cooled copper crucible. These alloys were deformed by rotary swaging of round bars from 13 mm to 9 mm diameter at a temperature of 1075 °C. Like Alloy 718, some alloys were produced by triple melting, i.e. Vacuum Induction Melting (VIM) + Electroslag Rapid Remelting (ESR) + Vacuum Arc Remelting (VAR). The ingot dimensions after the primary VIM step are 200 mm in diameter and appr. 650 mm in length. Forging was done similarly to standard Alloy 718. After wrought processes, all alloys were solution annealed at 980 °C/1.5 h, followed by water cooling to room temperature (RT). The material was then double aged as standard Alloy 718 at 721 °C/8 h + furnace cooled at 50 °C/h + 620 °C/8 h air cooled to RT.

For the examination of long term microstructural stability of the alloys, the heat treatments were conducted at a temperature of 700 °C for 500h and at 800 °C for 500 h and 2000 h.

For microstructural analysis by scanning electron microscopy (SEM) the samples were mechanically polished

and afterwards etched. Two etching media, namely V2A-stain and Canadas, were used in this research. When the V2A-stain was applied, the matrix was etched and all precipitations were not affected. The grain boundaries could be identified very well, too. Canadas was used in order to evaluate the amount and morphology of precipitations; the matrix and grain boundaries were not affected.

The chemical composition of V2A-stain is 200 ml H_2O , 200 ml HCl , 20 ml HNO_3 and 0.6 ml “Vogels Sparbeize”. The chemical composition of Canadas is 80 ml H_2O , 20 ml H_2SO_4 , 20 ml HF and 10 ml HNO_3 .

3. Alloy design

The new alloys have been designed to be (i) processed by normal industrial wrought processes and (ii) have superior microstructural stability at temperatures of up to 750 °C. In order to achieve (ii) the solvus temperature of the γ' phase must be increased so that its amount rises as well. That can be achieved by increasing the Al, Ti and Nb amounts in the material. However, a study of Cozar and Pineau reports that (Al + Ti + Nb) content should be lower than 7.5 at.% [10,11] because of processing considerations. All alloys with a higher (Al + Ti + Nb) content had problems during forging. Furthermore, the Nb content (5.4 wt.%) cannot be raised in the new alloy because of a tendency toward segregation during the melting and remelting processes with the resulting precipitation of brittle Laves phase, which is not acceptable in the material. The research by Cao and Kennedy on the development of Alloy 718Plus to date has tended to focus on (Al + Ti) [4,5] content rather than (Al + Ti + Nb) content for the same reason. They limited the (Al + Ti) content to 4.0 at.% and studied different Al/Ti (at.%) ratios. For Alloy 718Plus the best combination of mechanical properties and microstructural stability at higher temperature was observed at an Al/Ti ratio of about 4. However, not only the (Al + Ti) content with constant Nb content must be defined, but the highest γ' solvus temperature acceptable for forging as well: it must be lower than 1030 °C because otherwise it would be as difficult as Waspaloy forging. Increasing the amount of the γ' phase at constant Nb content particularly reduces the forging window. In order to be processed by normal industrial wrought processes (i) the forging window must be about 100 °C, and (ii) the precipitation kinetics have to be slower than in Waspaloy. The new materials should be free from brittle phases such as Laves, σ or η phases, and the microstructure of a new alloy must be stable after long term heat treatment at a temperature of 800 °C for 500 h. Co can be used for decreasing the γ' phase solvus temperature [12–14], and for this reason, Co was included in this study. Increasing the Co content to up to 15 wt.% during this study in Alloy 718 unexpectedly raised the solvus temperature of the δ phase by around 50 °C. Figure 1 shows the thermodynamic prediction of the phase's solvus temperatures for the solid phases and the solidus temperature for alloys with a continuously changing concentration of Co. The Co concentration was varied from 0 wt.% to 50 wt.%. The Ni concentration was balanced; the other elements stayed constant as in standard Alloy 718. Furthermore, as per ThermoCalc simulation, the Laves phase should be stable at a Co content of about

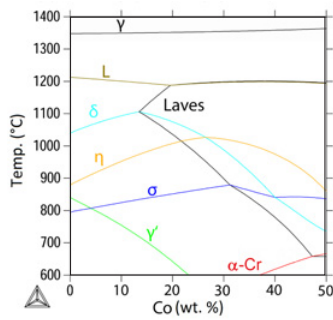


Figure 1. Quasi-binary Ni-Co diagram based on Alloy 718 where Ni is replaced by Co.

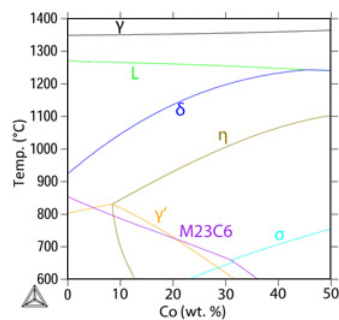


Figure 2. Quasi-binary Ni-Co diagram based on Alloy 718 without Fe (Ni is replaced by Co).

15 wt.% and above. Because Fe is usually responsible for the precipitation of the Laves phase in Alloy 718 it was excluded in order to destabilize the Laves phase in the alloys. The same simulation without Fe predicted an increasing stability of the δ phase with increasing Co content in the material, see Fig. 2. However, Co also stabilizes the η phase. If at 10 wt.% Co the η phase is stable in the small temperature range between 820 °C and 750 °C, at 30 wt.% Co the η phase will be stable at 1000 °C and below, according to diagram 2. The γ' phase will be destabilised with this precipitation of η phase. The solvus temperature and stability range of the σ phase also rises stably with an increasing Co concentration in the material.

Firstly, the stabilising effect of Co on the δ phase was studied. To do this, an alloy with 50 wt.% Co was melted. This alloy should have a primary δ phase. Figures 2a and 2b show the microstructure of alloy L13 with 50 wt.% Co and Alloy 718 after homogenisation. The δ phase in Alloy 718 (see Fig. 3a) can be partially observed at grain boundary triple points. The grains and grain boundaries are δ phase free. In contrast to Alloy 718 large particles of δ phase can be identified on the microstructure of L13 (see Fig. 3b). This is consistent with the ThermoCalc predictions.

During this investigation the Co range was mostly studied between 17 wt.% and 32 wt.%. The Co content has to be adjusted to the Ti and Al content because of the interaction between element content and precipitated phases such as δ , η and γ' phase.

In Fig. 4 the γ' phase solvus temperature of experimental alloys in dependence on (Al + Ti) content is presented. In order to fix the solvus temperature of this phase between 900 °C and 1030 °C (horizontal lines)

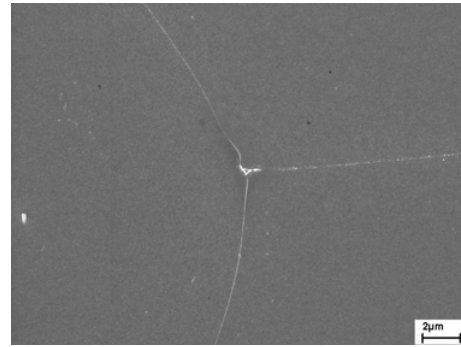


Figure 3a. δ phase on grain boundary triple point after homogenisation of Alloy 718.

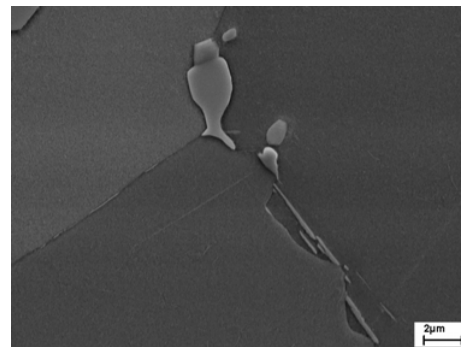


Figure 3b. δ phase after homogenisation of alloy L13 with 50 wt.% Co.

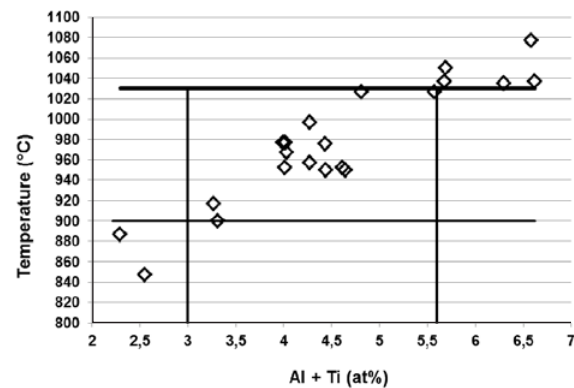


Figure 4. The variation of γ' phase solvus temperature in experimental alloys dependent on (Al + Ti) content.

a (Al+Ti) content between 3 at.% and 5.6 at.% (vertical lines) was chosen. The most interesting alloys for this study have a (Al + Ti) content between 4 at.% and 5 at.% and as a result the γ' phase solvus temperature is situated between 940 °C and 1000 °C. In order to produce a new alloy by normal industrial wrought processes the forging window should be about 100 °C to allow fine grain size forging. For this purpose, the Co content must be adjusted accordingly. In Fig. 5, the forging window of experimental alloys as a function of different Co and (Al + Ti) contents is presented. The vertical lines at 3 at% and 5.6 at% were taken from Fig. 4. In addition, vertical boundary lines at 3.7 at% and 4.7 at% limit the generally optimum (Al + Ti) content in new alloys. The horizontal line at 80 °C indicates the minimum forging window of new alloys. The

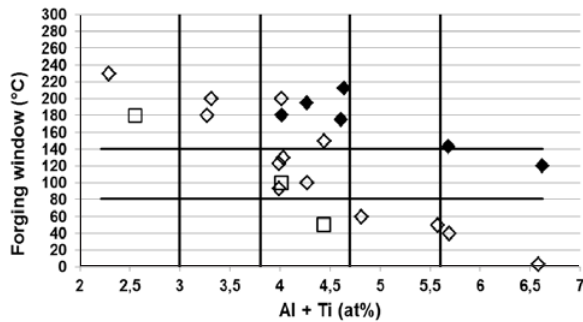


Figure 5. The forging window of experimental alloys in dependence on Co and (Al + Ti) contents; Co content: square-up to 11.5 at.%, white rhomb 11.5 at.% $\leq$ Co $\leq$ 18 at.%, black rhomb- more than 18 at.%.

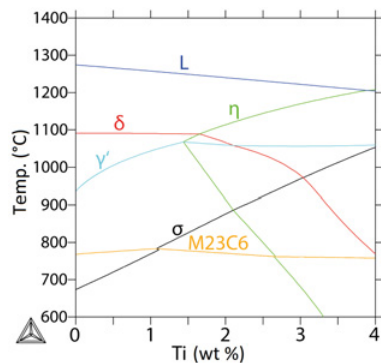


Figure 6. Quasi-binary Ni-Ti diagram with 17 wt.% Co, 2 wt.% Al, 5.4 wt.% Nb, 2.96 wt.% Mo, 18.9 wt.% Cr and 0.025 wt.% C (Ni is replaced by Ti).

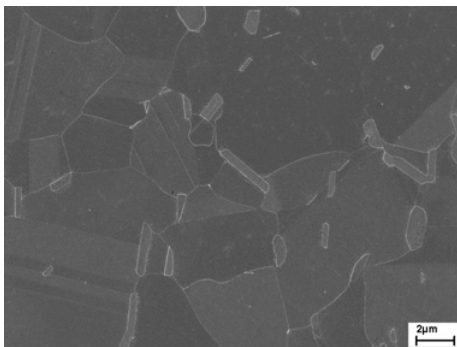


Figure 7. Microstructure of alloy L4 (pilot plant scale) after standard precipitation heat treatment.

horizontal line at 140 °C indicates the optimum forging window. It is clearly recognizable that the forging window decreases with increasing (Al + Ti) content but increases with the amount of Co content.

However, in Fig. 2 the precipitation of the η phase with increasing Co content was predicted in Alloy 718. It is well known that the stability of the η phase can be influenced by the Ti content in the material. As a consequence, not only the (Al+Ti) content must be defined, but the Al/Ti ratio as well. It was found that when the Co content in new alloys is increased, then the Ti content should be reduced. Consequently, the Al content must be raised because of the stability of the γ' phase. For instance, in Fig. 6 the

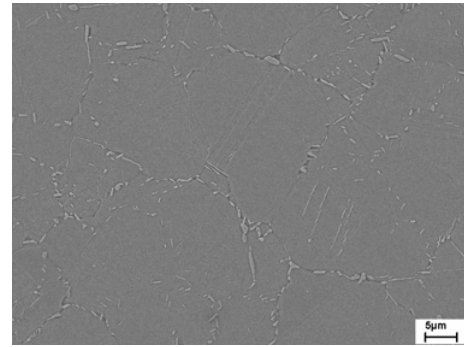


Figure 8a. Microstructure of alloy L4 (laboratory scale) after heat treatment: 700 °C/500 h.

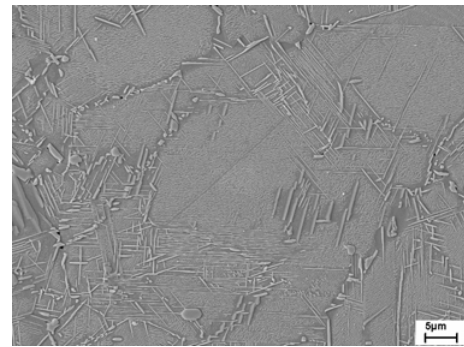


Figure 8b. Microstructure of Alloy 718 (laboratory scale) after heat treatment: 700 °C/500 h.

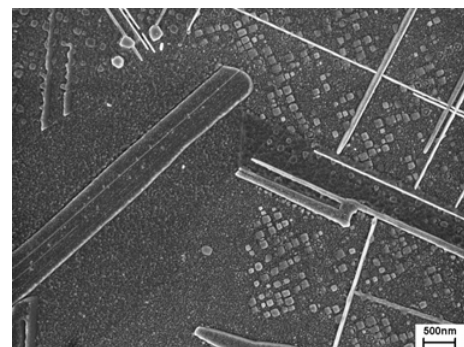


Figure 9. Microstructure of alloy L4 (laboratory scale) after heat treatment: 800 °C/500 h.

quasi-binary Ni-Ti diagram with fixed Co (17 wt.%), Al (2 wt.%), Nb (5.4 wt.%), Mo (2.96 wt.%), Cr (18.9 wt.%) and C (0.025 wt.%) content is presented. Two important parameters can be seen in this diagram: the changing size of the forging window by stabilising the γ' phase and the correlation between Ti content and precipitation of the η phase.

4. Results

Based on thermodynamic simulations, alloy L4 (Ni-17%Co-19%Cr-2.9%Mo-5.4%Nb-1.1%Ti-1.2%Al) was produced at pilot plant scale during this study. Because of the 17 wt.% Co the δ phase solvus temperature increased by about 50 °C to about 1080 °C. The higher content of Ti and Al influences the γ' solvus temperature. A small

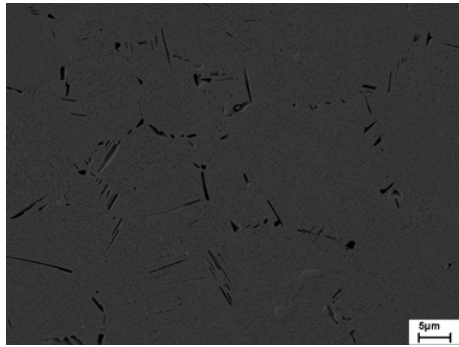


Figure 10a. Microstructure of alloy V16 (laboratory scale) after heat treatment: 800 °C/500 h.

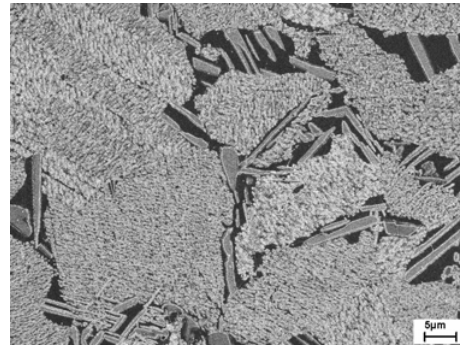


Figure 10c. Microstructure of alloy V16 (laboratory scale) after heat treatment: 800 °C/2000 h.

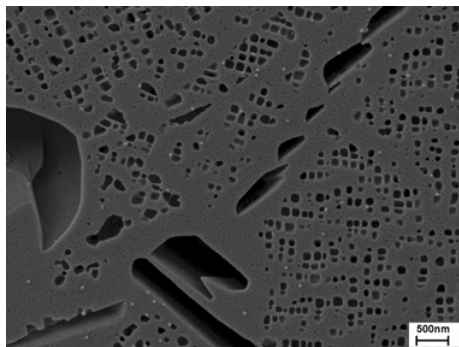


Figure 10b. Microstructure of alloy V16 (laboratory scale) after heat treatment: 800 °C/500 h.

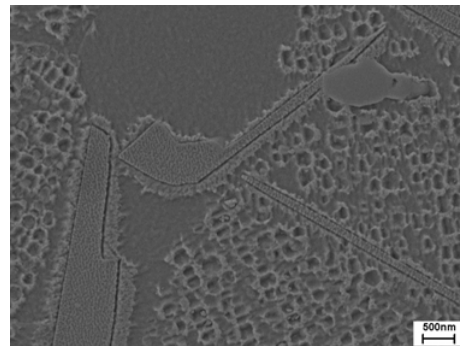


Figure 10d. Microstructure of alloy V16 (laboratory scale) after heat treatment: 800 °C/2000 h.

stability range of the η phase with volume fraction less than 3% was predicted by ThermoCalc in the temperature range between 940 °C and 980 °C. However, a η phase free microstructure was observed after standard precipitation heat treatment (see Fig. 7). The particles of the δ phase are dispersed on the grain boundaries. The microstructure is similar to that of Alloy 718. During this study delta forging with grain size ASTM 12 and finer also was done on pilot plant scale produced material. The alloy L4 has good microstructural stability at temperatures of up to 710 °C. Figures 8a and 8b demonstrate the microstructure of alloy L4 and Alloy 718 (laboratory scale) after long-time heat treatment at 700 °C/500 h. The microstructure of alloy L4 did not change while the microstructure of Alloy 718 changed significantly after the same heat treatment: the coarsening of γ'/γ'' and the precipitation of the plate-like δ phase in the grains can be observed.

However, increasing of the temperature up to 800 °C influences the microstructure of alloy L4 significantly: the plate-like η phase precipitates in the grains and the precipitates of γ'/γ'' can be seen (Fig. 9). It can be explained with the low Al/Ti ratio of 2.0 (at.%)

In order to achieve an η phase free microstructure at higher temperature and given Co content, the Al/Ti ratio must be adjusted. For this purpose, the alloy V16 (Ni-17%Co-19%Cr-2.9%Mo-5.4%Nb-0.2%Ti-2.0%Al) was chosen as an example for this paper. This alloy has a high Al/Ti ratio (20.0) and the sum of (Al+Ti) is 4.27 at.%. The forging window reaches 100 °C. This alloy shows an excellent microstructural stability at a temperature of 800 °C for 500 h and 2000 h (see Figs. 10a–d).

After 2000 h microstructure coarsening can be seen, but the microstructure is still η phase-free.

5. Summary and Conclusions

The role of the alloying elements Al, Ti, Fe and especially Co in 718-type alloys were studied using thermodynamic simulations and experimental evaluations with the aim of designing a new alloy family that (i) can be processed by normal industrial wrought processes and (ii) has superior microstructural stability at temperatures of up to 750 °C. The following conclusions can be drawn from this study:

- The addition of 15 wt.% Co and more to 718-type alloys significantly stabilises the δ phase. Its solvus temperature can be increased to such an extent that even primary precipitation from the melt may occur at extreme amounts of Co. To prevent this and to ensure good forgeability at the same time, the ideal Co range is 17 to 32 wt.%.
- As the solvus temperature of the δ phase is increased by Co addition, the solvus temperature of the γ' phase can be raised accordingly by a larger content of the γ' forming elements without compromising the forging window. However, it is necessary to select a high Al/Ti ratio at the same time.
- Co and Ti stabilise the η phase alike, even though the effect of Ti is much more pronounced. Thus, it is essential to increase the Al/Ti ratio to insure improved microstructural stability.

- The best combination of thermal stability and mechanical properties occurred at an (Al + Ti) level of about 4.2 to 4.8 at.% and an Al/Ti (at.%) ratio of more than 7. In order to achieve microstructural stability at even 800 °C the Al/Ti (at.%) ratio must be elevated to 15 or higher.
- A new superalloy family was developed during this study. These alloys have a forging window comparable to Alloy 718, but superior microstructural stability and thus allow for significantly larger service temperatures.
- A new alloy – VDM Alloy 780 Premium, with a defined chemical composition – selected from these detailed studies is actually in production starting with a 20 t primary VIM melt to produce VAR-ingots that will be forged into billets.

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