Metallographical and Thermodynamic Investigation of the Microstructures at 1200°C of TaC–Containing Co–Based Alloys

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ARTICLE INFO	ABSTRACT

Among the wide variety of current metallic structural materials able to work at elevated temperatures in severe chemical and mechanical conditions, some superalloys based on cobalt play a particular role: the ones rich in chromium for resisting hot corrosion by gases and melts and strengthened by tantalum carbides. They are able to demonstrate good behavior in contact of molten salts or glasses and simultaneously under mechanical stresses. Their refractoriness as well as the characteristics of their TaC population are key points for their sustainability at high temperature in working conditions. The present study aims to anticipate the long time microstructure behavior at high temperature by testing three similar cast Co–based superalloys strengthened by TaC carbides but different from each other in the field of the chemical compositions of their matrix. Thermodynamic calculations and long time exposure at 1200°C were carried out to predict their microstructure evolution at this very high temperature, with special focus of the influence on TaC carbides of the chemical compositions of the alloys, notably of their matrix.

INTRODUCTION

High-temperature materials are available in different types to allow various machines operating high temperatures. For instance, refractory ceramics are present in blast furnaces to slowly downwards drive the getting hotter alternate layers of iron-ore and coke as well as in drainage channels conveying separately the produced molten cast iron and slag (Cameron *et al.*, 2019). Because of different specifications than for ceramics (high mechanical properties for all solicitation modes, good toughness…), metallic materials are preferred for blades and disks of aero-engines, power generation turbines, or rotating tools for shaping molten glass. These later ones are superalloys, metallic materials with high melting points and exceptional resistance against high-temperature oxidation, corrosion, and creep deformation (Sims *et al.*, 1972).

The most used among these high-temperature alloys are the ones based on nickel, very efficiently strengthened by gamma prime coherent Ni3Al–type densely precipitated fine particles or by dispersed yttria nanoparticles, single-grained or with coarse orientated grains (Donachie *et al.*, 2002). Unfortunately, the first ones lose all their exceptional strength above 1100°C and the second ones are very hard to fabricate, and the mechanical behavior of both of them is rather anisotropic. Despite their conventional elaboration way (classical foundry, but in an inert atmosphere) equiaxed polycrystalline superalloys, although their mechanical behavior is – at about 1000°C and below – generally significantly weaker than the previously evocated alloys, can keep strength up to 1200°C, and even slightly above. This is, for example, the case of cobalt-based superalloys containing MC–type carbides in a quantity high enough: TaC (Michon *et al.*, 2003), HfC (Berthod *et al.*, 2014), TiC (Khair *et al.*, 2016), NbC and ZrC (Berthod, 2017).

Among the different types of MC carbides, the most common is tantalum carbide. It can be found among the various carbides present in old cobalt alloys, for instance, SM-302 (Felten *et al.*, 1964) and WI-52 (Eng *et al.*, 1972). Much more recently, studies revealed the great interest in having a

carbides network exclusively made of TaC. This can be simply achieved by choosing atomic contents for tantalum equal to the carbon one, or slightly higher. The absence of any other carbides (e.g. chromium carbides) allows maintaining the melting start temperature of the alloy close to and even higher than 1300°C (Berthod *et al.*, 2003), which is of great importance for good mechanical behavior at around 1200°C. When conventionally cast it crystallizes only at the end of solidification, forming a eutectic compound with the matrix. The script–like morphology of these obtained primary TaC carbides is very helpful for interdendritic strength at elevated temperatures. However, for achieving high mechanical performances in hot conditions these script–like carbides must be present in quantities high enough (typically carbon content > 0.2 wt.%) and for avoiding dramatic loss in toughness carbides must be not in a too-large fraction (typically carbon content < 0.8 wt.%). In addition to conditions related to volume fractions of carbides, another point of watchfulness is keeping the TaC single–phased character of the carbides population. Indeed, depending on the composition of the alloy, and particularly of the matrix, other carbides may be present even if there are as many Ta atoms as C atoms. When it is respected this criterion leads to carbides being only TaC in nickel–free cobalt-based alloys, even rich in chromium, (Michon *et al.*, 2003) while it is not sufficient for chromium-rich nickel-based alloys (Berthod *et al.*, 2004).

Since cobalt-based superalloys often contain more or less nickel for different purposes – crystalline network stability for matrix (Sims *et al.*, 1972; Donachie *et al.*, 2002; Bradley, 1988) or chromium volume diffusion easiness (Kofstad, 1988) – the uniqueness of the carbide nature (only TaC) and an optimized TaC volume fraction make that well controlling the carbide fraction is of great importance. This is this control that will be done here for three different alloys with various nickel contents. Since thermodynamic modeling is also a potentially helpful mean for anticipating both nature and fraction of carbides, the comparison will be made and, in case of good agreement between the two ways – experimental and modeling – thermodynamic calculations will be used to explore microstructures at 1200°C beyond the Ni, Cr, Ta and C contents of the three alloys.

METHODS
Alloys and samples
Three compositions of alloys were considered: (a) For the "Cob1" alloy: 25 wt.%Cr, 0.4 wt.%C, and 6 wt.%Ta. (b) For the "Cob2" alloy: same contents in Cr, C, and Ta as "Cob1" but the addition of 13.7 wt.%N. (c) For the "Cob3" alloy: same contents in Cr, C, and Ta as "Cob1" and "Cob2", but more Ni: 27.4 wt.%

Taking the high-temperature reactivity of C, Cr, and Ta (introduced as pure elements, as Co and Ni) in oxygen-containing gases, melting, high-temperature liquid state (five minutes), and cooling were performed in an inert atmosphere of pure argon (pressure: about 0.3 atm). The obtained ingots (ovoid compact shape, mass of about 40 grams), after their complete cooling and extraction out of the 50kW–furnace (CELES, France), were cut using a metallographic saw into several parts. A part was taken to prepare a metallographic sample (embedded mirror-like section for the as-cast microstructure characterization) following the classical procedure, and another one to prepare a sample for high-temperature exposure.

High-temperature exposure
Three samples, each coming from one of the three ingots, were placed in alumina nacelles which were introduced in the hottest zone of a resistive high-temperature muffle furnace (NABERTHERM, Germany). They were exposed at 1200°C for a week. After this high-temperature exposure, they were cut and prepared for metallographic characterization as the first sample.

Characterization of the as-cast and 1200°C–aged microstructures
The microstructures of the three alloys, in their as-cast state and after 1200°C–aging, were observed with a Scanning Electrons Microscope (SEM; model: JEOL JSM-6010A, JAPAN), in Back Scattered Electrons mode (BSE). The chemical compositions of the alloys were controlled by full-frame Energy Dispersion Energy (EDS) with the EDS spectrometer attached to the SEM. The chemical composition of the matrix in the 1200°C–aged state was measured with the same spectrometer but by spot analysis.

Thermodynamic modeling
The theoretical metallurgical states of the three alloys corresponding to their chemical compositions, the 1200°C temperature, and the 1

atm pressure were computed using the N version of the Thermo–Calc software and the SSOL thermodynamic database preliminarily enriched with the descriptions of additional subsystems involving tantalum: Co–Ta (Liu *et al.*, 1999), Ni–Ta (Ansara *et al.*, 1994), Cr–Ta (Dupin *et al.*, 1993), Cr–Ni–Ta (Dupin *et al.*, 1996), Ni–Ta–C (Yuwen *et al.*, 1999) and Ta–Co–C (Dumitrescu *et al.*, 2001).

RESULTS AND DISCUSSION
The situation of the three alloys in the Co-Ni–Cr–Ta–C system

The study began with preliminary thermodynamic calculations aiming to the verification the natures of their microstructures, to see whether it can be hoped that their chemical compositions may lead to the desired {matrix + TaC} double–phased state at the working temperature, and also whether the TaC may be of a eutectic nature (with some chance to be really mixed with matrix and to be also of the Chinese script morphology). A first diagram (Figure 1) was

computed with Thermo – Calc and the Ta–enriched SSOL database. This is an isopleth section of the quinary diagram with fixed contents in Cr (25wt.%), Ta (6wt.%), and C(0.4wt.%), and temperature and Ni content (at the expense of cobalt) as varying parameters.

This isopleth section, which extends from the liquid domain down to rather low temperatures, is rather complex. There are many different domains due to the allotropic FCC ↔ HCP transformation of the cobalt-based matrix, the two types of chromium carbides (Cr7C3 and Cr23C6), and the possibility of an appearance of the Ni3Ta intermetallic compound at low temperature. One can point out the influence of nickel which tends to favor the FCC crystalline network of the matrix (decrease in transition temperature with higher Ni content) and which also favors the Cr7C3 carbides at the expense of TaC. Adding Ni also lowers a little both solidus and liquidus temperature.

Figure 1. Isopleth section at {25 wt.%Cr, 0.4wt.%C and 6 wt.%Ta} of the {Co, Ni, Cr, C, Ta} system, calculated with Thermo–Calc; position of the 3 alloys
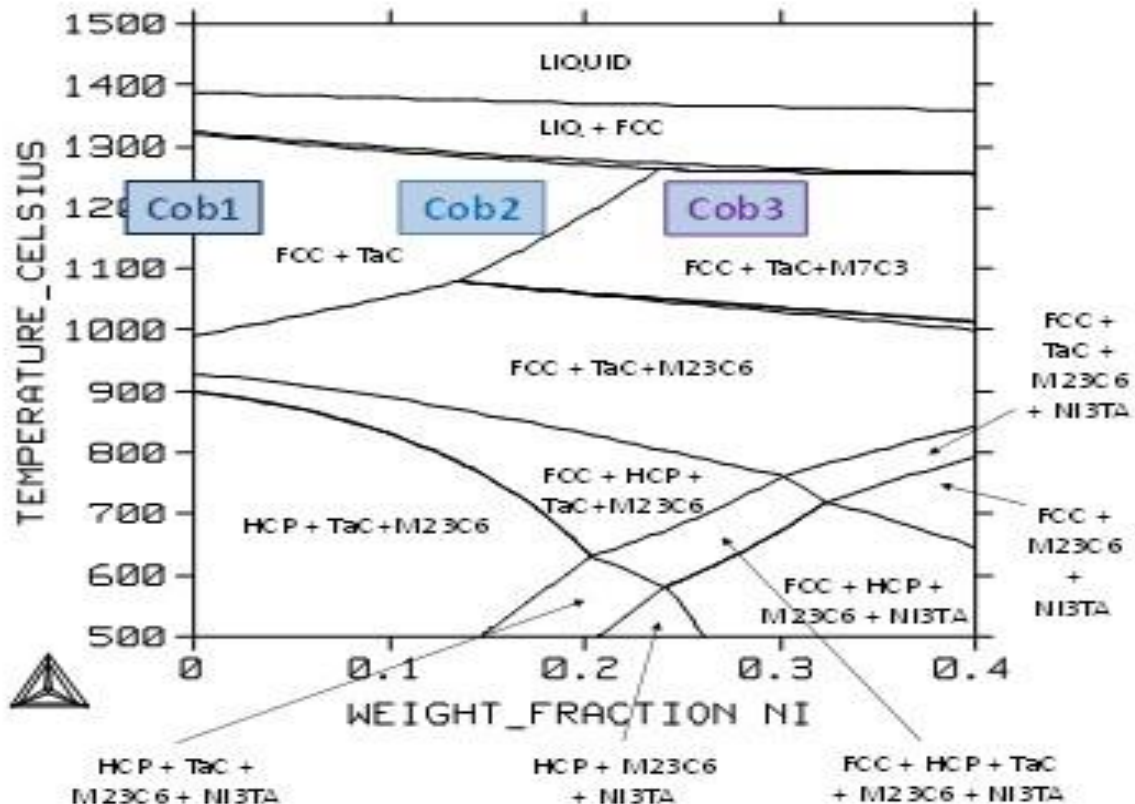
According to this section of the diagram, at 1200°C, the "Cob1" and "Cob2" alloys seem to be

effectively composed of matrix and TaC carbides only. Furthermore, when temperature decreases

from the liquid state, both alloys would go down crossing a {liquid, matrix} double–phased domain, before crossing a thin {liquid, matrix, TaC} triple–phased domain for finishing their solidification. It can be expected that their microstructures will be composed of a dendritic matrix, the dendrites of which are separated from one another by a {matrix, TaC} compound. The "Cob3" alloy risks are slightly different. Indeed, its solidification ought to proceed as for the two first alloys but it is finished by the isothermal precipitation of a double–phased {matrix, Cr7C3} or triple–phased {matrix, TaC, Cr7C3} eutectic compound.

Another view of the positions of the three alloys is given in the 1200°C–isothermal section presented in Figure 2), which is simpler than the previous figure to clearly see the expected microstructures of the three alloys at this temperature. One can easily see that the microstructure of Cob1 should be really double–phased {matrix, TaC}, even if there are some small variations in Ta; Cob2 should be of the same microstructure but Cr7C3 may appear if the obtained tantalum content is a little too low. Cob3 risks containing Cr7C3 in addition to the TaC. To avoid that 1 wt.%Ta more ought to be enough.

Chemical compositions and microstructures (as–cast and aged at 1200°C)

The chemical compositions of the obtained alloys, as specified by EDS, are presented in Table 1. The wished contents in Ni and Cr are well respected. This seems to be different for the Ta contents; in fact, the obtained values are a little overestimated.

Figure 2. 1200°C–section for {25 wt.%Cr and 0.4wt.%C} of the {Co, Ni, Cr, C, Ta} system with varying contents in Ni(Co) and Ta, calculated with Thermo–Calc; positions of the 3 alloys
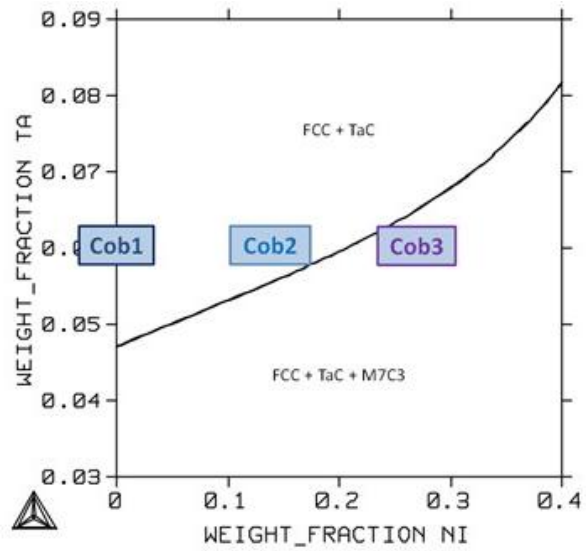
This is due to the fact that Ta is mainly concentrated in TaC carbides and the ones of these carbides which emerge on the polished surface interact more with the electron beam, inducing an over-representation of the Ta element. So, on can consider that no Ta was lost during the foundry operation and thus that the real Ta contents in the alloys are really 6 wt.%. Concerning carbon, a very light element and additionally present in the alloys with very low concentration, its content cannot be measured by EDS.

The obtained microstructures after casting are shown in Figure 3: left micrographs for Cob1, middle micrographs for Cob2, and right micrographs for Cob3. As suspected before, the as-cast Cob1 and Cob2 alloys are really {matrix, TaC} double–phased.

Table 1. Compositions of the elaborated alloys (5 full frames ×250 EDS analyses → average value ± standard deviation value)

All contents in wt.%	Cob1	Cob2	Cob3
Co	Balance		
Ni	/	13.1 ±0.4	25.5 ±0.2
Cr	25.6 ±0.1	26.5 ±0.4	25.7 ±0.2
Ta	7.5 ±0.1	7.5 ±0.2	7.6 ±0.2
C	Supposed to be 0.4 (well respected)		

Furthermore, as expected, the matrix is dendritic and the TaC carbides (white/bright

particles) are located in the inter–dendrites spaces where they are mixed with matrix, forming a eutectic compound without any doubt. The morphology of these TaC carbides is obviously Chinese–script. The Cob3 alloy looks like the two first alloys but one can notice, here and there, some small very dark particles: Cr7C3 carbides. EDS spot analysis demonstrated local very high contents in Ta and in C in the bright particles and high contents in Cr and in C in these small dark particles. However EDS spot analysis results staid only qualitative since all these particles were too small and the interaction peer-produced by the electron beam ran over them and reached a part of the neighbor matrix too.

These microstructures were partly lost during the long exposure at 1200°C. Even if the carbide distribution staid globally the same (interdendritic) the script–like morphology of the TaC is replaced by alignments of more or less coarse round carbides. The driving force of this geometrical change was simply the minimization of the interfacial energy associated with the matrix–TaC boundaries.

Compositions of the matrixes after 1200°C–exposure; comparison with Thermo–Calc

After about a full week isothermally spent at 1200°C, each of the three alloys has reached its thermodynamic equilibrium. This one is necessarily characterized by specific values of mass fractions in matrix and TaC and by specific chemical compositions of both phases. The chemical composition of the matrix was analyzed by EDS for the three alloys and the results are displayed in Table 2. In parallel calculations performed with Thermo–Calc gave another version (theoretical) of the same data. They are added to Table 2 too, for comparison. Good agreement is found between measurements and calculations for the contents in Ni, Cr, and Ta, globally. This suggests that the Ta–enriched SSOL database can allow Thermo–Calc to give good predictions.

Figure 3. As–cast (top) and 1200°C–aged (bottom) microstructures of the Cob1 (left), Cob2 (middle) and Cob3 (right); SEM/BSE ×1000 micrographs
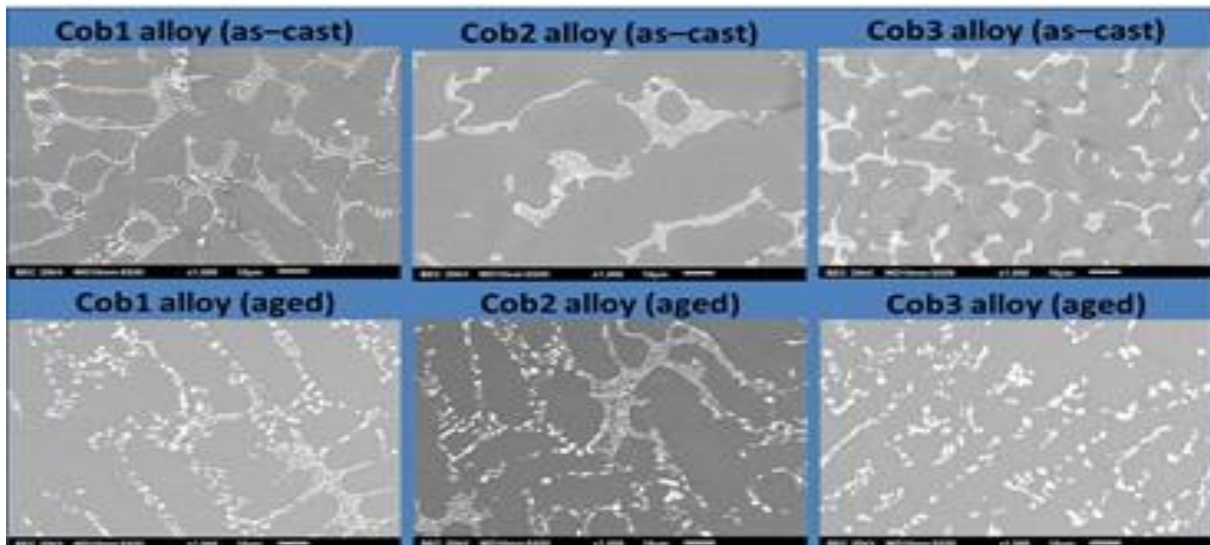
TaC mass fractions and volume fractions

From these results of Ta contents in the matrix, it is possible to deduce the mass fractions of the obtained TaC, since the difference in Ta content between alloy (6 wt.%Ta) and matrix (close to 2 wt.%, according to EDS results) corresponds to the tantalum part involved in the formation of the TaC carbides. Thus, the mass fractions of TaC carbides can be estimated according to the first following equation, and their volume fraction can be deduced using the second following equation:

$$\%_{TaC}{}^{m} = \{M_{TaC}\times(\%_{Ta}{}^{all}-\%_{Ta}{}^{mat})\}/\{M_{Ta}-M_{TaC}\times\%_{Ta}{}^{mat}\} \qquad (1)$$

$$\%_{TaC}{}^{v}= \{\%_{TaC}{}^{m}/\rho_{TaC}\}/\{(1-\%_{TaC}{}^{m})/\rho_{mat}+\%_{TaC}{}^{m}/\rho_{TaC}\} \qquad (2)$$

$\%_{TaC}{}^{m}$: mass fraction of TaC,

M_{TaC} : molar mass of TaC (globally 193 g/mol),

$\%_{Ta}{}^{all}$: Ta weight content in the whole alloy (6 wt.%),

$\%_{TaC}{}^{mat}$: Ta weight content in the matrix (EDS measurements),

M_{Ta} : molar massof Ta (181 g/mol),

$\%_{TaC}{}^{v}$: volume fraction of TaC,

ρ_{TaC} : volume mass of the TaC carbide (14.3 g/cm^3),

ρ_{mat} : volume mass of the matrix (supposed to be close to 9 g/cm^3).

The results are presented in Table 2. The mass fractions of TaC according to Thermo–Calc calculations were converted into volume fractions using equation (2) too. For the volume fractions resulting from the EDS measurement – equation (1) and conversion using equation (2) – three values were considered: the one obtained for the average value of Ta weight content in matrix issued from EDS, the one obtained from this average value-added with the standard deviation, and the one obtained from the average value to which standard deviation was subtracted. It appears that the TaC volume fraction is around 3vol.% for both the calculated results and the experimental ones. Here too there is thus good agreement between thermodynamic calculations and real experiments. Figure 4 allows comparison between the initial SEM/BSE images and the digitalized ones with grey threshold rating leading to the surface fractions equal – or at least very close – to the volume fraction of Table 3 (EDS, "average"). The digitalized images well represent the SEM/BSE images in grey levels: this demonstrates that the values obtained by both experimental measurements and thermodynamic calculations are realistic.

Table 2: TaC volume fractions issued from Thermo–Calc calculations, and TaC volume fractions deduced from the EDS–measured Ta contents in the matrixes of the 1200°C–aged alloys (1: average - sd, 2: average, 3: average Ta + sd; sd: standard deviation)

MATRIX at 1200°C (wt.%)	Cob1	Cob2	Cob3
Thermo-Calc (deduced from this mass.%)	3.31 (5.22)	3.29 (5.20)	3.00 (4.75)
1200°C–aged vol.% TaC from wt.%Ta in matrix (EDS)	3.59 [1] **2.65** [2] 1.69 [3]	3.19 [1] **3.06** [2] 2.92 [3]	3.19 [1] **3.06** [2] 2.92 [3]

Figure 4. Comparison between the initial SEM/BSE images and the black & white digitalized ones with ratings giving the same fractions as in Table 2
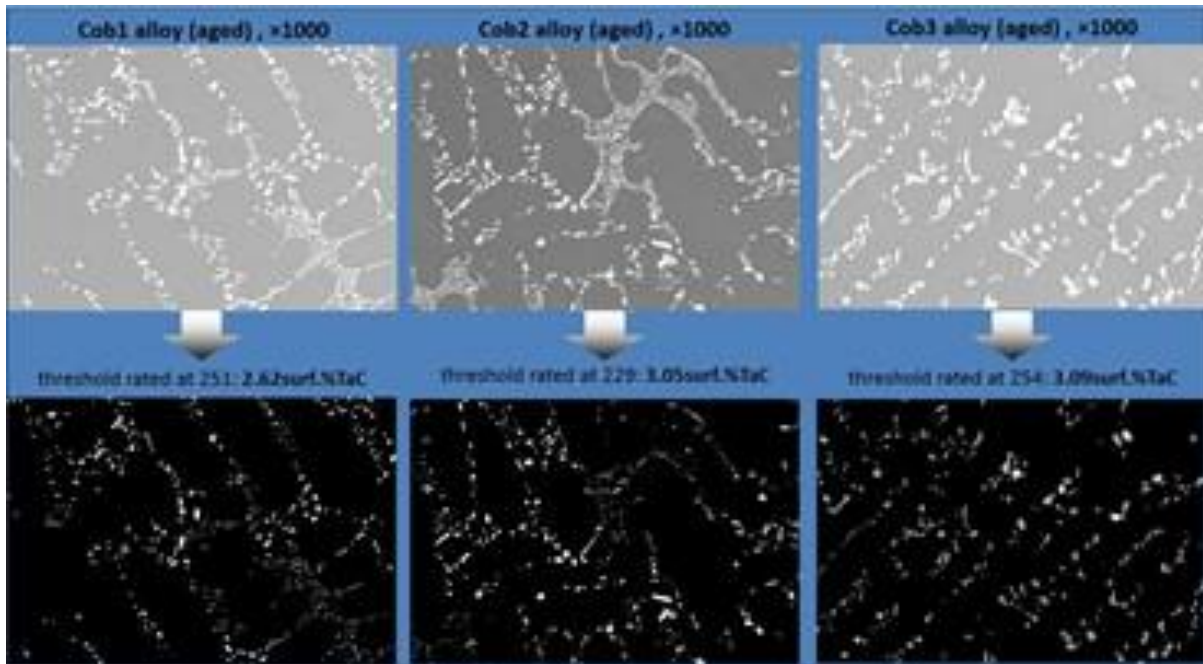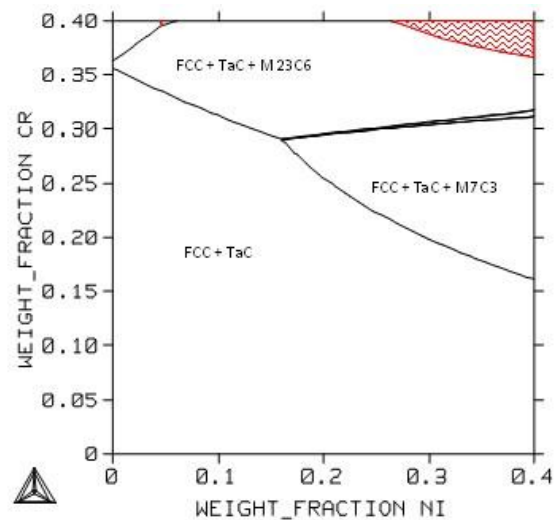

Microstructures at 1200°C beyond the limits of the three alloys explored by calculations

The metallurgical states calculated for the three alloys at 1200°C by Thermo–Calc and the enriched SSOL database were relevant, as demonstrated by the good agreement with the experimental results. It was therefore decided to take benefit from this modeling tool to explore the microstructures derived from the ones of the three alloys, by modifying simultaneously the contents in Ni (at the expense of Co), Cr, Ta, and C by pairs: {Ni, Cr}, {Ni, Ta} and {Ni, C}.

The microstructures at 1200°C of Co–Ni–Cr–Ta–C alloys when the Ni and Cr contents both vary for fixed contents in Ta (6 wt.%) and C (0.4wt.%) are presented in the isothermal section given in Figure 5. The main part of the {0 to 40 wt.%Ni} × {0 to 40 wt.%Cr} square corresponds to the double–phased {matrix, TaC} state. Increases in Ni and in Cr, separately or simultaneously, finish provoking the appearance of chromium carbides, Cr23C6 for low Ni contents and high Cr contents, and Cr7C3 for higher Ni contents and lower Cr contents. For 0.4 wt.%C and 6 wt.%Ta, the Cr threshold to do not exceed for keeping a double–phased {matrix, TaC} state at 1200°C is globally 35 wt.%Cr in absence of nickel, slightly less than 25 wt.%Cr if 20 wt.%Ni is present, and slightly more than 15 wt.%Cr if the alloy contains 40 wt.%Ni (the

alloy is then more Ni-based than Co–based in such situation. Since a minimal Cr content of about 25 wt.% is compulsory to allow the alloy being chromia–forming (i.e. able to efficiently resist high-temperature oxidation and corrosion by developing and maintaining a protective external chromia oxide scale), the Ni addition cannot exceed 20 wt.%Ni if it is wished that TaC remains the single carbide present. One must also mention the possible start of melting for contents in Ni and Cr too high simultaneously (top-right corner of the section).

Figure 5. Metallurgical states at 1200°C for varying contents in nickel and chromium

The microstructures at 1200°C are simpler for the {0 to 40 wt.%Ni} × {0 to 10 wt.%Ta} composition field (Figure 6). By maintaining the Cr content at 25 wt.%, the Ta one at 6wt.%, and the C one at 0.4 wt.%, the double–phased {matrix, TaC} state corresponds to a trapeze delimited by the (0wt.%Ni, 5wt.%Ta), (40wt.%Ni, 8wt.%Ta), (40wt.%Ni, 10wt.%Ta) and (0wt.%Ni, 10wt.%Ta) points. From this domain, a decrease in Ta may lead to the appearance of a second carbide phase: Cr7C3. The Ta limit increases from about 4.7wt.%Ta for 0wt.%Ni to slightly more than 7wt.%Ta for 35wt.%Ni, for instance. Ni tends to destabilize TaC for too low Ta contents. For much lower Ta contents, TaC may be absent and only Cr7C3 exists. For instance, for 40wt.%Ni, slightly more than 2.5 wt.%Ta may be present in the matrix only, in a solid solution. In the total absence of Ni, only 0.7 wt.%Ta allows such a situation. Clearly, Ni favors Cr7C3 at the expense of TaC.

Figure 6. Metallurgical states at 1200°C for varying contents in nickel and tantalum
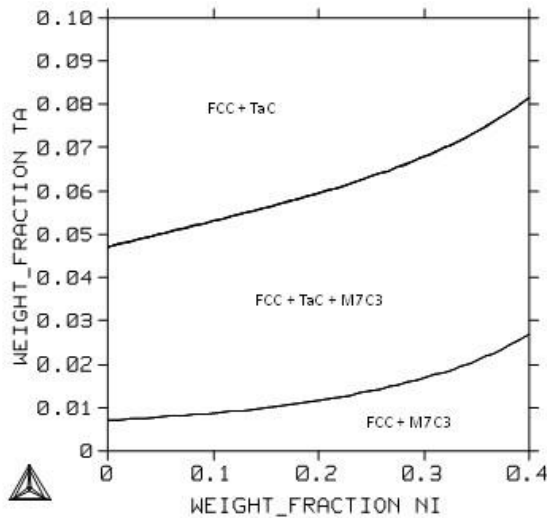
When the Cr and Ta contents are fixed at 25 wt.%Cr and 6 wt.%Ta, variations in C weight content govern the nature of the carbide population (Figure 7). With moderate C content it is possible to keep, at 1200°C, the double–phased {matrix, TaC} microstructure (globally the bottom half of the {0 to 40 wt.%Ni} × {0 to 1 wt.%C} composition field (except the too low C contents band where carbides can be absent and where alloys can be partly melted). More C addition promotes the appearance of chromium carbides. Here too nickel demonstrates its deleterious effect on the double–phased {matrix,

TaC} state. The higher the Ni content, the lower the C content to preserve the {matrix, TaC} microstructure.

Fig. 7: Metallurgical states at 1200°C for varying contents in nickel and carbon
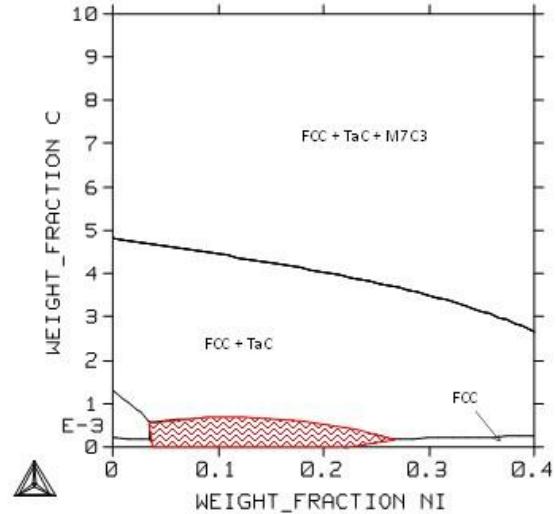
CONCLUSION

Many MC carbides are recognized to allow high mechanical performances at elevated temperatures, due to their particular morphology and their high stability, high stability which intervenes upon solidification and bereaves chromium to form carbides… if the MC–forming metal is present with content high enough for the available C quantity. Tantalum is one of the most frequent atoms present in superalloys, either to harden the matrix by solid solution effect (due to its size) or to favor the presence of carbides – TaC generally – if the superalloy contains carbon. Obtaining TaC carbides, even in presence of a rather high quantity in chromium (added for hot oxidation and corrosion reasons), is easy in cobalt-based alloys but elements such as nickel, and alloying elements are often present in the compositions of Co-based superalloys to improve different properties and behaviors, may disadvantage TaC. With the three alloys studied here, which respect the {at.%Ta = at.%C} rule, TaC was the single carbide present, furthermore well-shaped (script morphology), well–located (interdendritic spaces) and in rather high quantity (more or less 5 mass.% and 3 vol.%), regardless of the Ni content (up to almost 30wt.%Ni). However, thanks to thermodynamic calculations carried out, after having verified their accuracy with the three

alloys experimentally characterized, the possible presence of additional chromium carbides was revealed – and with them, refractoriness lowering – which must be taken into consideration. The calculation of isothermal sections showed that, with the third alloy, the chemical composition was almost critical and chromium carbides can easily appear in case of a carbon content a little too high, a tantalum content a little too low, or a chromium content a little too high. Fortunately, the same isothermal sections show how modifying the chemical compositions prevents obtaining such not desired microstructures.

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