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# Volume Fractions of Tantalum Carbides Deduced from the Ta Contents in the Matrix of Three 1250°C–Aged Cobalt–Based Alloys; Comparison with Thermodynamic Calculations

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## Abstract

Some superalloys for service at high temperature under stresses are strengthened by tantalum carbides. Their creep resistance depends on the quantity of TaC and this is the reason why it is often important to control the volume fraction of these carbides in the microstructure. Metallographic preparation followed by electron imaging and surface fraction measurements by image analysis is a frequent way for that. Another possibility is to deduce the mass fraction of TaC, and after their volume fraction, from the chemical composition of the matrix when the alloys are only double-phased, on the {matrix + TaC} type. In this work three alloys – chemically designed to be made exclusively of matrix and TaC – were elaborated and isothermally exposed to an elevated temperature for a duration long enough to allow the alloys being at their thermodynamic equilibria. The chemical compositions of the alloy and of its matrix were measured and the results allowed evaluating their TaC mass fractions which were converted in volume fractions. The obtained TaC fractions were compared to results issued from thermodynamic calculations. Good agreement was found for the three alloys, and this allowed to exploit the used software and thermodynamic database to explore further the microstructures at the same high temperature, notably to know the conditions on the Co, Ni, Cr, Ta and C contents to keep the {matrix + TaC} structure and to avoid any possible partial melting.

**Keywords:** Co-based alloys; Elevated temperature; TaC volume fraction; Thermodynamic Calculations.



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## 1. Introduction

Carbides were among the early first metallurgical solutions to confer sufficient mechanical properties to cast alloys for uses at very high temperature in conditions of mechanical stresses statically or periodically applied. Since the first alloys to be used in the hottest parts of aircraft turbines were rich in chromium to combat both high temperature dry oxidation and corrosion by melts [1], chromium carbides – easily obtained in alloy with such a chromium-rich chemical environment, played this useful reinforcing role [2]. With the increasing performances of the aeronautic turbines resulting from higher operating temperatures in the combustion cans, the mechanical resistance of superalloys reinforced by chromium carbides started to be inadequate [3]. Among the reasons of that, there were the lack of geometrical stability of these carbides and their volume fractions lowered by the exposures at these higher temperature levels. Beside other strengthening metallurgical ways, the carbide-strengthening solutions started to involve new carbides, more stable at elevated temperature, TaC carbides for example. This led to cobalt-based alloys such as Mar-M 509 for instance, in which significant fractions in TaC co-exist with chromium carbides [4]. SM-302 [5] and WI-52 [6] are also cobalt-based superalloys containing Ta and in which MC carbides are present in the microstructure. Directionally solidified versions of these superalloys also existed [7]. Nowadays MC carbides in cobalt-based alloys are still under interest [8], and TaC carbides in particular [9].

The quantity of tantalum carbides on long term in a cobalt-based superalloys is one of the main factors governing the mechanical behavior at elevated temperature. Even in case of presence of TaC with significant volume fraction just after elaboration, the complexity of the chemical composition may induce a decrease in their quantity, even a total disappearance of this phase in the microstructure after more or less long stay at high temperature [10]. It can be thus of importance to test the microstructures at high temperature and to control the stabilized TaC fractions. This may be by exploiting further X-Ray Diffraction patterns, by image analysis on metallographic samples.

In this work three alloys based on cobalt and containing TaC carbides with great fractions to candidate to high mechanical performances at elevated temperature, were subjected to the evaluation of the remaining quantities of, their TaC carbides, by another method, easier to use. This simply exploited the Ta contents in the matrix which, by assuming that the tantalum quantity which is not in the matrix is necessarily involved in carbides. This allows first knowing the mass fractions of TaC carbides and therefore their volume fraction, and if the measured Ta contents in matrix are close to the ones calculated by a thermodynamic equilibria solver tool, this one mechanically leads to the good mass or volume fractions. The Thermo-Calc software working with a database designed to well represent the equilibrium states of the alloys of the studied family, will be tested. In case of good predictions it will appear as a possible useful tool to track other sources of risk of mechanical weakening in addition to carbides disappearance: the proximity of a liquid state.

## 2. Material and Method

### 2.1. Names Chemical Compositions and Fabrication of the Considered Alloys

The three alloys are all based on cobalt and they were all wished to contain 25 weight percent of chromium to resist oxidation by hot gases as well as corrosion by hot aggressive liquid substances. They were designed to respect the atomic equivalence between carbon and tantalum to promote the formation of TaC carbides exclusively (no more carbon atoms than tantalum atoms in order to avoid the formation of chromium carbides). Knowing that 0.4 wt.% of carbon is a good compromise between the need of efficient carbides-strengthening and the necessity to maintain a good toughness, the tantalum content was rated to fit this carbon content: same atomic content between C and Ta, as is to say a weight content in Ta equal to fifteen times the C one (i. e. 6 wt.% Ta for 0.4 wt.%C). A fifth element was added to two of the three alloys – nickel – with two contents: about 14 wt.%Ni and about 27 wt.%Ni. This element, often added to cobalt-based alloys to control their tendency to change of crystalline network in case of thermal cycling (Hexagonal Compact to Face Centered Cubic and inversely), may have also an influence of the TaC stability and of the refractoriness of the alloys.

The “CCC” alloy, Co(bal.)–25Cr–0.4C–6Ta, the “CCn” alloy, Co(bal.)–14Ni–25Cr–0.4C–6Ta, and the “Cnn” alloy, Co(bal.)–27Ni–25Cr–0.4C–6Ta, were all produced as 40 grams–weighing compact ingots, from pure elements (purity > 99.9%, provider: Alfa Aesar) and using a high frequency induction furnace (manufacturer: CELES, France). Heating, melting, 5 minutes–stay in the liquid state, liquid state cooling, solidification, solid state cooling were done in an inert atmosphere made of 300–500 mbars of pure argon.

### 2.2. Microstructure and Chemical Characterization

Each ingot was cut to obtain, on the one hand, a sample for the examination of its as-cast microstructure and, on the other hand, a sample to expose at high temperature. The first one was embedded in a cold resin system (resin and hardener from ESCIL, France), ground (SiC papers from 240 to 1200#, water-washed and ethanol-dried, and polished (1µm-hard particles on a rotating textile disk) until a mirror-like state was obtained. The obtained metallographic sample was submitted to electronic examination using a Scanning Electron Microscope (SEM) in Back Scattered Electrons (BSE) mode to control, and to Energy Dispersion Spectrometry (EDS), to control the obtained microstructures and the chemical composition, respectively. The second sample was ground with 1200# SiC paper and exposed to heat during 70 hours at 1250°C constantly. After return to ambient temperature this second sample was prepared as described before in the case of the as-cast sample. Its microstructure was examined using the SEM in BSE mode to observe the microstructures evolution of during the long stage at elevated temperature, in particular to verify if the microstructure – certainly modified – is still of the same type as in the as-cast condition: double-phased with matrix and TaC carbides only. EDS spot analysis were carried out in the matrix (assumed to be chemically homogeneous after the stabilization at 1250°C). Five EDS measurements were performed and the results led to an average value and a standard deviation one.

### 2.3. Comparison with Thermodynamic Calculations; Determination of the TaC Fractions

The Thermo-Calc software (version N) and a home-made thermodynamic database (the SSOL database enriched with the description of several sub-systems involving tantalum: Co – Ta [11], Ni – Ta [12], Cr – Ta [13], Cr – Ni – Ta [14], Co – Ta – C [15] and Ni-Ta-C [16] was used to predict the metallurgical state of the three alloys at 1250°C from their chemical compositions, and notably to know the chemical composition of the matrix, its Ta content in particular.

The Ta contents in the matrix were exploited to determine the mass fractions of TaC carbides, according to the first one of the two following equations. The corresponding TaC volume fraction was deduced from the TaC mass fraction according to the second equation.

$$f_{\text{TaC}}^{\text{mass}} = \{M_{\text{TaC}} \times (g_{\text{Ta}}^{\text{all}} - g_{\text{Ta}}^{\text{mat}})\} / \{M_{\text{Ta}} - M_{\text{TaC}} \times g_{\text{Ta}}^{\text{mat}}\} \quad (\text{eq.1})$$

$$f_{\text{TaC}}^{\text{vol}} = \{f_{\text{TaC}}^{\text{mass}} / \rho_{\text{TaC}}\} / \{(1 - f_{\text{TaC}}^{\text{mass}}) / \rho_{\text{mat}} + f_{\text{TaC}}^{\text{mass}} / \rho_{\text{TaC}}\} \quad (\text{eq. 2})$$

In these equations,  $f_{\text{TaC}}^{\text{mass}}$  is the mass fraction of TaC,  $M_{\text{TaC}}$  is the molar mass of TaC (globally 181g/mol + 12g/mol = 193 g/mol),  $g_{\text{Ta}}^{\text{all}}$  is the Ta weight content in the whole alloy (6 wt.%),  $g_{\text{Ta}}^{\text{mat}}$  is the Ta weight content in the matrix (determined by EDS),  $M_{\text{Ta}}$  is the molar mass of tantalum (181 g/mol),  $f_{\text{TaC}}^{\text{vol}}$  is the mass fraction of TaC,  $\rho_{\text{TaC}}$  is the volume mass of the TaC carbide (14.3 g/cm<sup>3</sup>) and  $\rho_{\text{mat}}$  is the volume mass of the matrix (estimated at 9 g/cm<sup>3</sup>).

### 2.4. Exploration of the 1250°C–Isothermal Section for Different Couples of Varying Elements

In case of representativeness good enough of the thermodynamic predictions new calculations will be possibly carried out to exploit this tool for exploring the metallurgical states of alloys derived from the three ones for more extended variations of pairs of elements taken among Ni, Cr, Ta and C (with Ni at the expense of Co). Such calculations may play the role of very useful guidelines for the design of alloy, by indicating in which [wt.%E<sub>min</sub>, wt.%E<sub>max</sub>] × [wt.%E'<sub>min</sub>, wt.%E'<sub>max</sub>] frames – with (E, E') ∈ {Ni} × {Cr, Ta, C} – the {matrix + TaC} double-phased state exists at 1250°C (possible service temperature of some of these alloys), and which (E, E') combinations are to avoid (absence of TaC, partial presence of liquid...).

### 3. Results and Discussion

The alloys were successfully elaborated and cutting did not reveal the existence of not molten parts. Samples resulting from ingots cutting were ready for further investigations and tests: EDS and microstructure control and exposure at elevated temperature.

#### 3.1. Obtained Chemical Compositions and Microstructures

The chemical compositions of the three alloys are displayed in Table 1, well fit with the targeted contents in nickel and chromium. The tantalum contents are, as usual when EDS is used for such type of alloys, a little overestimated: the real Ta contents, necessarily slightly lower than the values given by EDS, are in good agreement with what was wished. Concerning the carbon content, it cannot be specified by EDS because this element is rather light and, in addition, present in too low quantity in the alloys. It is supposed to be correctly respected, by considering the density of the obtained TaC population.

**Table-1.** Chemical compositions of the obtained alloys (average  $\pm$  standard deviation from five full frame  $\times 250$  EDS analyses); \*: carbon cannot be analyzed; it is supposed to be well respected

Alloys ( $\times 250$ ) wt.%	CCC	CCn	Cnn
Co	Bal.	Bal.	Bal.
Ni	/	13.1 $\pm$ 0.2	25.5 $\pm$ 0.4
Cr	25.2 $\pm$ 0.5	26.2 $\pm$ 0.3	25.7 $\pm$ 0.5
Ta	7.7 $\pm$ 0.5	7.9 $\pm$ 0.4	7.7 $\pm$ 0.2
C	0.4*	0.4*	0.4*

The microstructures of the alloys in their as-cast states are illustrated by SEM/BSE micrographs in Figure 1. They are all double-phased, composed of a dendritic matrix and tantalum carbides precipitated in the interdendritic spaces, obviously during a eutectic reaction transforming the residual liquid in a second matrix part and TaC carbides closely imbricated.

These alloys were exposed at 1250°C for 70 hours. Figure 2 shows how microstructures have evolved during this heat exposure. Obviously TaC fragmentation and coalescence modified a little the carbides network.

#### 3.2. Qualitative Comparisons with Calculated Theoretical Microstructures

The three alloys are positioned in the isopleth section of the {Co, Ni, Cr, C, Ta, temperature T} diagram calculated with Thermo-Calc for the {25wt.%Cr, 0.4wt.%C, 6 wt.%Ta, pressure P = 100 kPa}–fixed conditions (Figure 3). According to this figure, the “CCC” and “CCn” alloys would be composed at 1250°C of matrix and TaC phase only. The “Cnn” alloy should contain also Cr<sub>7</sub>C<sub>3</sub> carbides, but in much lower quantity than TaC as suggested to its position much closer to the double-phased {matrix + TaC} domain than to the {matrix + M<sub>7</sub>C<sub>3</sub>} one. In addition one can also remark that 1250°C is just a little lower than the solidus temperature. The same observations (double-phased or triple-phase states, proximity of solidus) can be done in 1250°C–isothermal sections such as in Figure 4 where Ni (at the expense of Co) and Ta vary at constant T, P, other contents.

**Figure-1.** As-cast microstructures of the three alloys

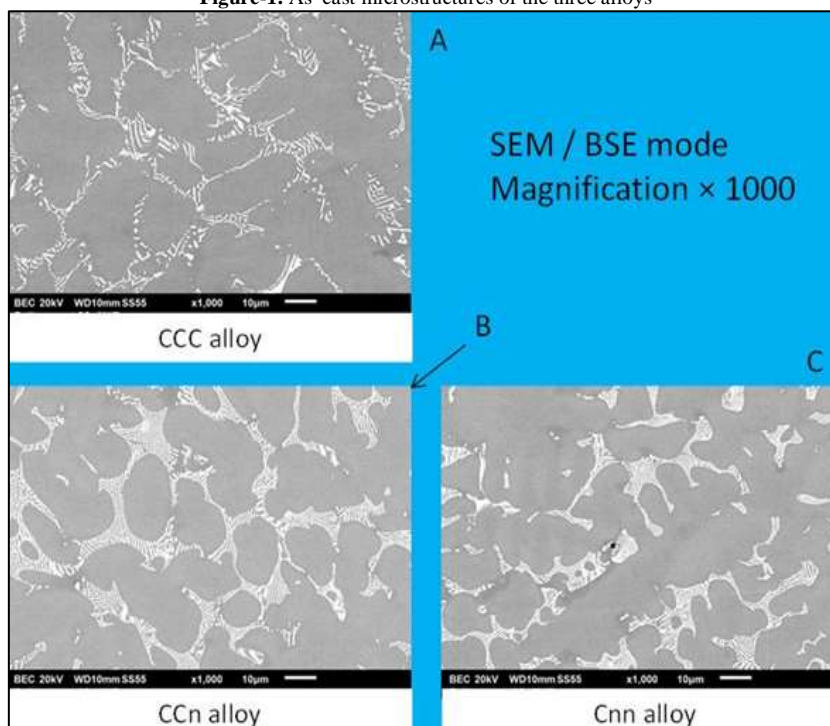


Figure-2. Microstructures of the three alloys aged at 1250°C

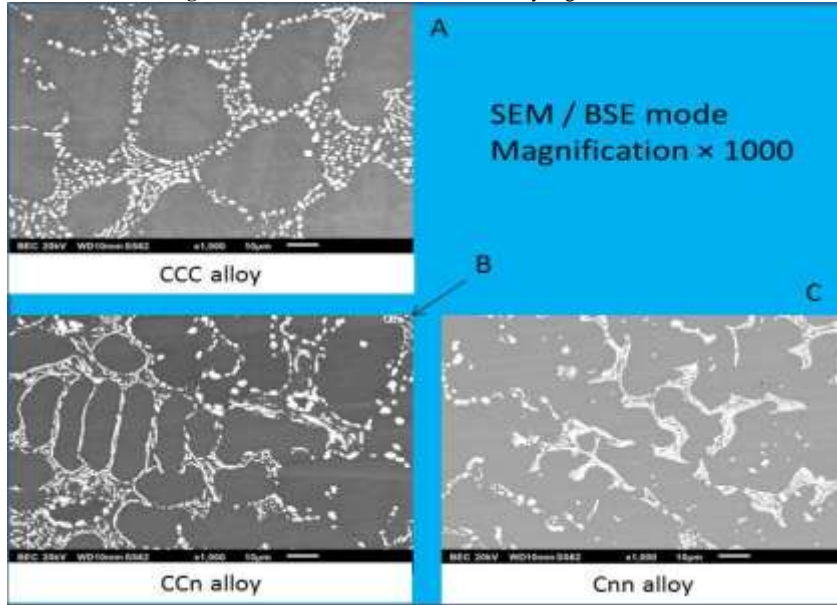


Figure-3. Positions of the three alloys in the isopleth section at {25 wt.%Cr, 0.4wt.%C and 6 wt.%Ta} of the quinary {Co, Ni, Cr, C, Ta} system; diagram computed with Thermo-Calc

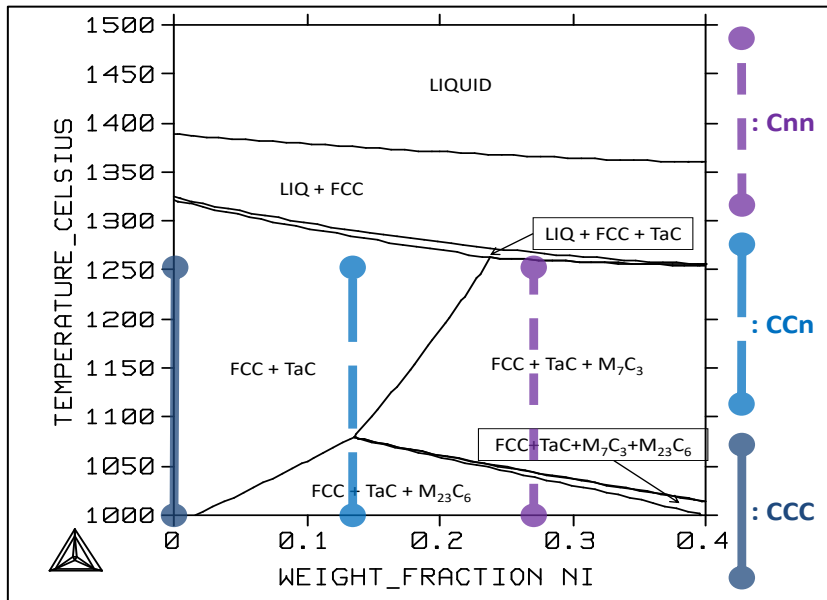
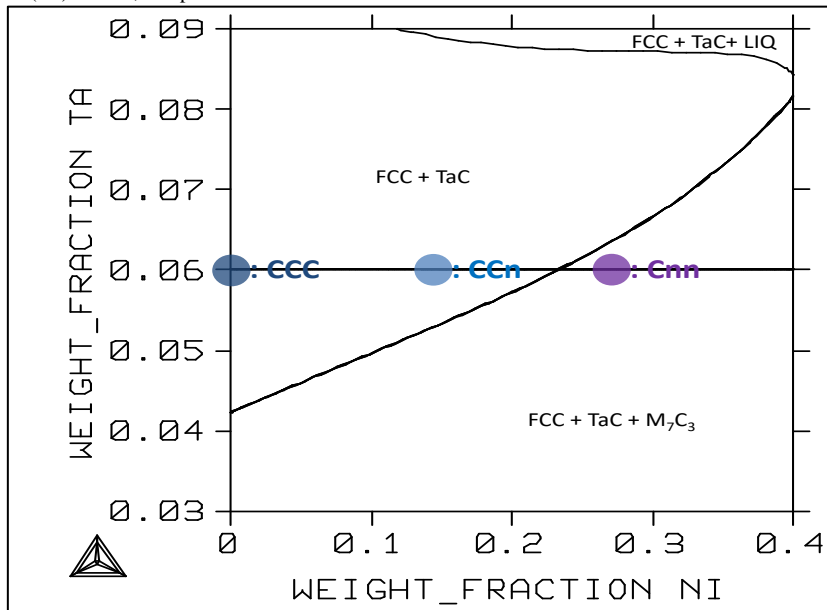


Figure-4. Positions of the three alloys in the 1250°C–isothermal section at {25 wt.%Cr and 0.4wt.%C} of the quinary {Co, Ni, Cr, C, Ta} system with varying contents in Ni(Co) and Ta, computed with Thermo-Calc



### 3.3. Quantitative Comparisons with Calculated Theoretical Microstructures

Table 2 gathers the chemical compositions of the matrix of the three alloys after homogenization and thermodynamic stabilization at 1250°C, according to Thermo–Calc calculations and really measured by EDS spot analysis. There is very good agreement between the two sets of values. This is notably true for tantalum for which the calculated Ta content is, in the three cases, located in the intervals defined by average Ta content  $\pm$  standard deviation.

**Table-2.** Comparison of the calculated and EDS–measured chemical compositions of the matrixes of the 1250°C–aged alloys (EDS: average  $\pm$  standard deviation from five full frame  $\times 250$  analyses)

Matrix at 1250°C wt.%	CCC	CCn	Cnn
Ni (Th-C)	/	14.44	28.85
Ni (EDS)	/	14.1 $\pm$ 0.3	27.2 $\pm$ 0.4
Cr (Th-C)	26.30	26.29	26.02
Cr (EDS)	27.0 $\pm$ 0.3	27.7 $\pm$ 0.3	27.2 $\pm$ 0.2
Ta (Th-C)	1.40	1.42	1.78
Ta (EDS)	1.6 $\pm$ 0.2	1.5 $\pm$ 0.2	1.6 $\pm$ 0.2
C (Th-C)	0.09	0.09	0.09
C (EDS)	Cannot be measured by EDS		

The calculated Ta weight contents as well as the measured ones were interpreted in term of mass fractions of TaC carbides according to (eq.1), which were converted into volume fractions according to (eq.2). The obtained volume fractions are given in Table 3. In the case of the volume fractions coming from the EDS results, three values were deduced: one for the average measured Ta content, one for this average value with standard deviation added, and one for the average value with the standard deviation subtracted. In each case the TaC volume fraction derived from the calculated TaC mass fraction is located between the two experimental extreme values. There is thus also good agreement between experimental and calculations.

**Table-3.** Comparison of the volume fractions of TaC deduced from the mass fractions calculated by Thermo–Calc, with the TaC volume fractions deduced from the mass fractions deduced from the EDS–measured Ta contents in the matrixes of the 1250°C–aged alloys (1: average Ta in matrix -  $\sigma$ , 2: average Ta in matrix, 3: average Ta in matrix +  $\sigma$ ;  $\sigma$  = standard deviation)

Alloys TaC surf.%	CCC	CCn	Cnn
Thermo-Calc (deduced from this mass.%)	3.17 vol.% (5.00 mass.%)	3.15 vol.% (4.98 mass.%)	2.92 vol.% (4.62 mass.%)
1250°C–aged vol.% TaC	3.19 vol.% <sup>(1)</sup> 3.06 vol.% <sup>(2)</sup>	3.26 vol.% <sup>(1)</sup> 3.13 vol.% <sup>(2)</sup>	3.19 vol.% <sup>(1)</sup> 3.06 vol.% <sup>(2)</sup>
from wt.% Ta in matrix (EDS )	2.92 vol.% <sup>(3)</sup>	2.99 vol.% <sup>(3)</sup>	2.92 vol.% <sup>(3)</sup>

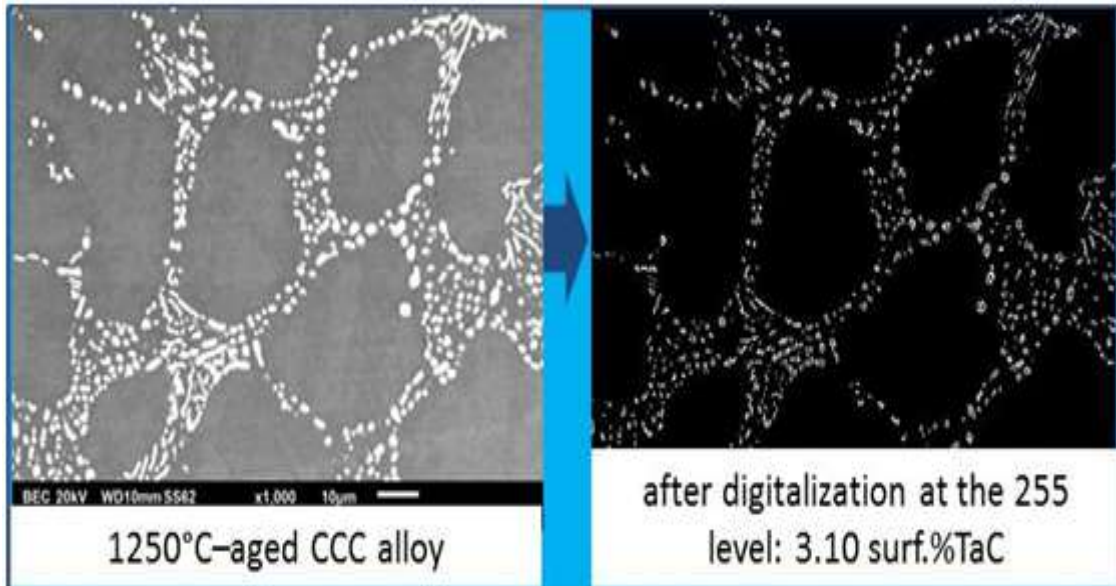
### 3.4. Visualization of the Deduced Volume Fractions

To verify whether these values of volume fractions well represent the TaC population as it can be seen by metallographic observation some examples of SEM/BSE micrographs were treated to become groups of black or white pixels depending on the grey level of each pixel of the micrographs. This was done by rating the grey level threshold in order to reach the previously volume fractions of TaC, and finally by comparing the initial micrograph and the binarized one. Results are given in Figure 5 for the “CCC” alloy, in Figure 6 for the “CCn” alloy and in Figure 7 for the “Cnn” alloy. It appears that, by choosing the threshold which allows obtaining surface fractions of TaC (white pixels) close to the volume fractions resulting from the conversion of the mass fractions deduced from Ta contents in matrix measured EDS – or from calculated mass fractions since calculated and EDS–deduced mass fractions are very close to one another – the figures formed by the white pixels are globally superimposed.

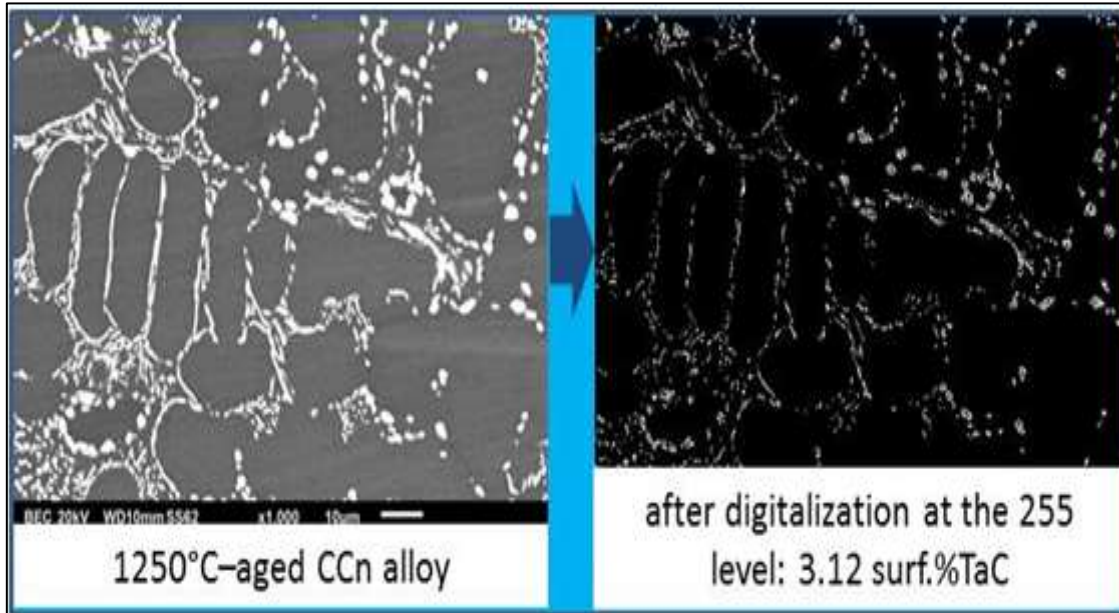
### 3.5. Exploration of the Predictable Variations in Microstructure at 1250°C

The calculations performed by Thermo–Calc associated to the home–made thermodynamic database have shown a good representativeness of the real microstructures at 1250°C of three alloys. It was decided to use this thermodynamic modeling tool to study the consequences of more or less limited changes in the weight contents of Ni, Cr, Ta and C. Three 1250°C–isothermal sections of the quinary system were computed. The section obtained by varying the Ni and Cr contents simultaneously is given in Figure 8, the one obtained by simultaneously varying the Ni and Ta contents is given in Figure 9 and the one with the contents in Ni and C varying at the same time is given in Figure 10.

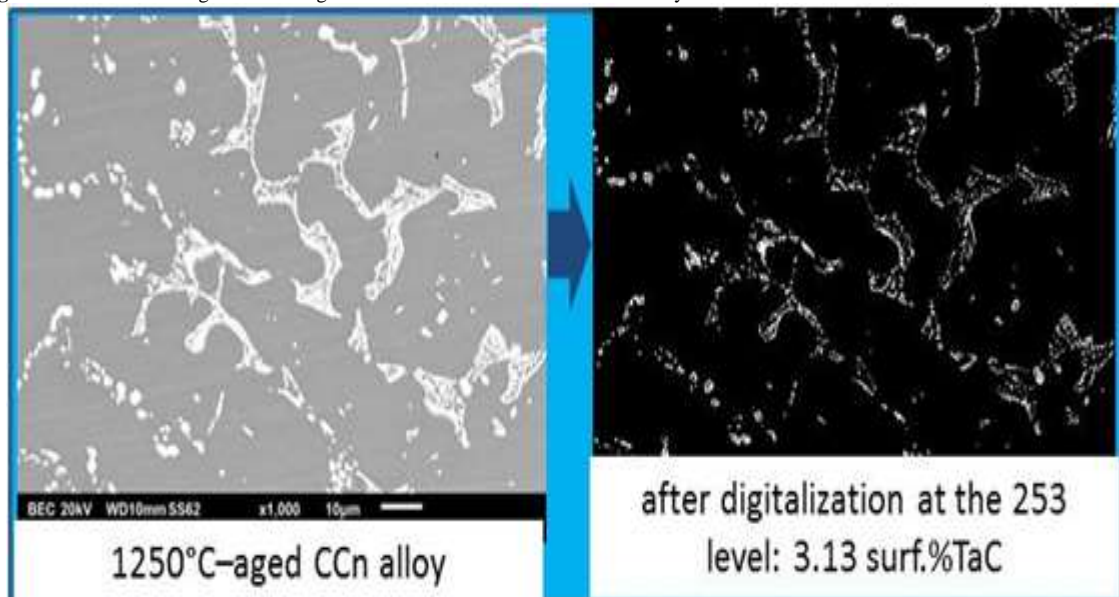
**Figure-5.** Threshold rating for retrieving the TaC surface fraction in the CCC alloy deduced from the EDS–measured Ta content in matrix



**Figure-6.** Threshold rating for retrieving the TaC surface fraction in the CCn alloy deduced from the EDS–measured Ta content in matrix



**Figure-7.** Threshold rating for retrieving the TaC surface fraction in the CCn alloy deduced from the EDS–measured Ta content in matrix



**Figure-8.** 1250°C–isothermal section at {25 wt.%Cr and 0.4wt.%C} of the quinary {Co, Ni, Cr, C, Ta} system with varying contents in Ni(Co) and Ta (up to 10 wt.%) computed with Thermo–Calc with the positions of the three alloys; proximity with a {solid + liquid} domain, notably for the Cnn alloy

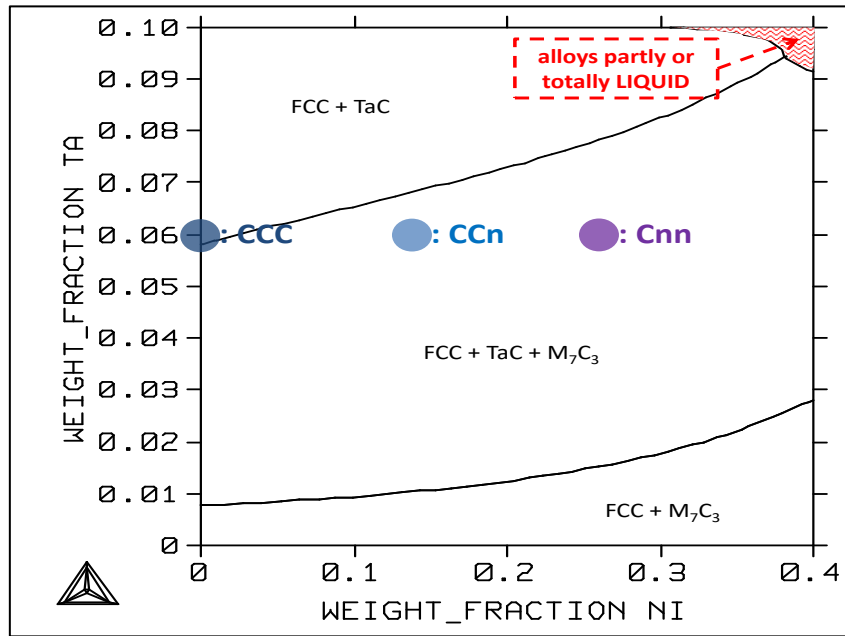
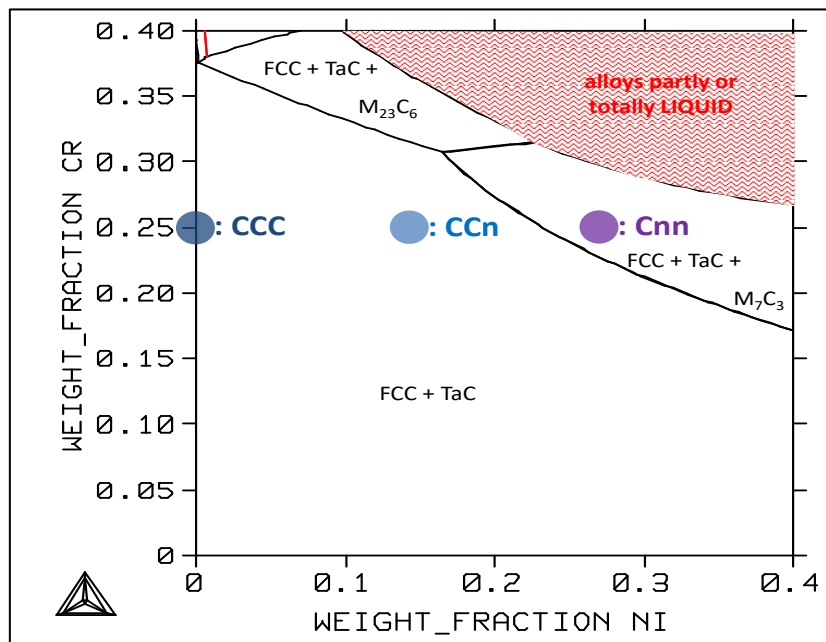


Figure 8 shows clearly that, for 1250°C, any increase in Ta content favors the existence of TaC carbides at the expense of the  $Cr_7C_3$  carbides, and also that adding more and more nickel inversely favors  $Cr_7C_3$  at the expense of TaC. In absence of nickel – and for 0.4 wt.%C – the boundary between the {matrix + TaC +  $Cr_7C_3$ } domain and the {matrix + TaC} one is stuck on 6wt.%Ta (Ta content threshold being very close to 5.8wt.%Ta, more precisely). This well corresponds to the atomic equivalence between C and Ta. But, when more and more nickel is present, the need in Ta for preserving the double-phased {matrix + TaC} state is increased. For instance, for 30wt.%Ni, 8wt.%Ta or more is compulsory for maintaining this {matrix + TaC} microstructure. On the low Ta side, the Ta content threshold of TaC appearance is about 0.8wt.%Ta in absence of Ni, but this threshold increases with the Ni content. For instance, when the alloy contains 40 wt.%Ni, at least 3 wt.%Ta are required to allow the presence of first TaC carbides in addition to the  $Cr_7C_3$  carbides. To finish with Figure 8, one can remark that in case of simultaneous high contents in Ni (e.g. 40 wt.%) and in Ta (e.g. 10 wt.%) the alloy ought to be partly molten at 1250°C. Increases in Ni and in Ta seem decreasing the refractoriness of the alloy.

In Figure 9 there is a wide domain where the alloys are double-phased {matrix + TaC} at 1250°C. This is only above the line joining the (0 wt.%Ni, 37 wt.%Cr) and (40 wt.%Ni, 17wt.%Cr) points, that chromium carbides may be present in addition to the TaC carbides:  $Cr_{23}C_6$  on the left (Co-rich side) and  $Cr_7C_3$  on the right (Ni-richer side). In case of contents in Ni and Cr simultaneously too high, liquid may be present.

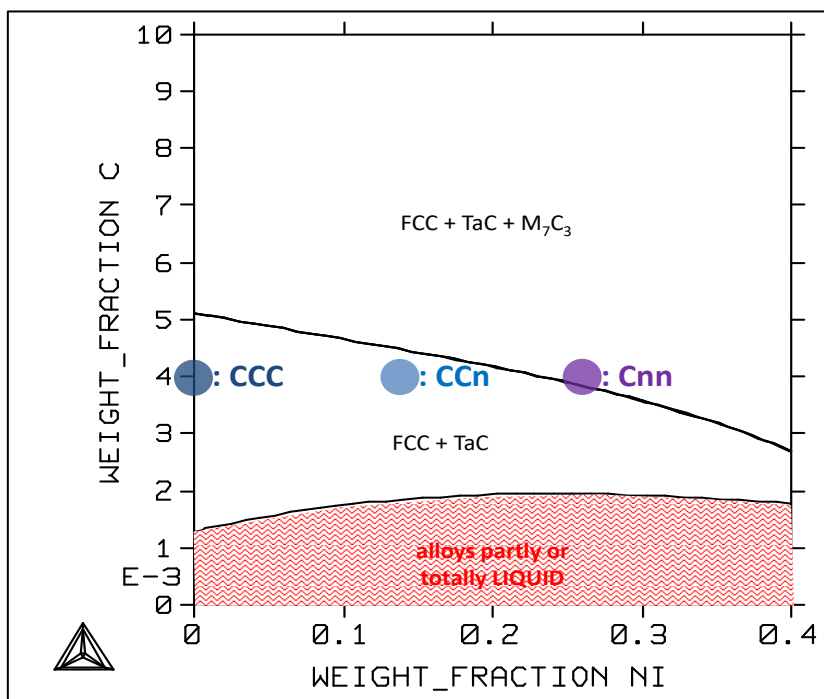
**Figure-9.** 1250°C–isothermal section at {6 wt.%Ta and 0.4wt.%C} of the quinary {Co, Ni, Cr, C, Ta} system with varying contents in Ni(Co) and Cr (up to 40 wt.%) computed with Thermo–Calc with the positions of the three alloys; proximity with a {solid + liquid} domain, notably for the Cnn alloy





In Figure 10, for given contents in Cr (25wt.%) and Ta (6wt.%) the carbon content appears as influencing factor for both the carbides population type and the alloy's refractoriness. High carbon promotes the formation of  $\text{Cr}_7\text{C}_3$  in addition to TaC while, for low C contents, the carbon atoms are monopolized by tantalum atoms, which leave nothing to chromium. Carbon seems favoring the refractoriness of the alloys. This was not really expected and thus a little surprising since carbon favors carbides which are of a eutectic type for carbon contents not too high.

**Figure-10.** 1250°C–isothermal section at {25 wt.%Cr and 6 wt.%Ta} of the quinary {Co, Ni, Cr, C, Ta} system with varying contents in Ni(Co) and C (up to 1 wt.%) computed with Thermo–Calc with the positions of the three alloys; proximity with a {solid + liquid} domain, notably for the Cnn alloy



#### 4. Conclusion

The knowledge of the quantity of carbides able to stay present at high temperature in the microstructure of a superalloy, the mechanical strength of which is based on hardening by these particles, is of great importance to predict the lifetime of components made of this superalloy. In the case of conventionally cast carbides-strengthened cobalt-based superalloys, exploiting the high morphological stability of primary tantalum carbides is well recognized solution to allow uses for rather long times at elevated temperature. However TaC carbides must be present with volume fractions high enough, not only in the as-cast condition but also at the service temperature. Verifications can be done on artificially high temperature aged superalloys but metallography limited to imaging can be not accurate enough. Furthermore it can lead to dangerous overestimation of the TaC fractions, because of their very bright tint when observed in electron microscopy. In this work it was attempted to deduce TaC volume fractions from TaC mass fractions, themselves deduced from Ta contents in matrix. The obtained results demonstrate that this way is rather efficient. In addition, the experimentally obtained values of Ta content in matrix for thermodynamic equilibrium at 1250°C allowed testing a home-made database designed to be exploited by Thermo–Calc. The good correspondence between measured values and calculated values showed that the database was rather accurate for predicting microstructures at 1250°C. This allowed carrying out different types of calculations / isothermal sections of diagrams, potentially helpful for choosing alloy's composition for reaching the wished microstructures and for avoiding partial molten states.

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