Experimental and Thermodynamic Study of Tantalum-containing Iron-base Alloys reinforced by Carbides: Part II-case of (Fe, Ni, Cr)-base austenitic steels

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Abstract. Experiments and thermodynamic calculations were performed for three alloys based on both iron and nickel in equal quantities, containing 30wt.% of chromium, 0.2 to 0.4wt.% of carbon and 3 to 6 wt.%Ta of tantalum. Solidus and liquidus temperatures were determined for the three alloys, and natures and fractions of the existing phases at 1000, 1100 and 1200°C were characterized. These experimental data were compared to values calculated by Thermo-Calc, as well as to such results obtained on previous similar alloys separately based on iron or on nickel. Generally there is a good agreement with calculations. Tantalum carbides in (Fe,Ni) base alloys are less present than in Fe-base alloys, but more present than in Ni-base alloys for the same carbon, chromium and tantalum contents.

Keywords: Iron/nickel-based alloys, Tantalum carbides; Chromium carbides; Experimental characterization; Thermodynamic modeling

1. Introduction

The high temperature mechanical properties of cast superalloys can be achieved by precipitation of primary or secondary carbides, respectively during solidification and during an hardening heat treatment [1,2]. Notably, chromium carbides may exist in corrosion-resistant chromia-forming superalloys. When tantalum also exists in the chemical composition of the alloy, tantalum carbides may also appear. These TaC carbides are candidates to strengthen superalloys up to very high temperatures. Indeed, they are very refractory and are stable in significant amounts in cobalt-base superalloys for example [3], while they are not stable in other systems such as nickel-base alloys as seen earlier in some nickel alloys [4]. Tantalum carbides can also be present in great quantities in ferritic iron-base alloys, even if their microstructure may evaluate when exposed to very high temperatures and long times [5].

The topic of this work is to explore, as previously done on Fe-30Cr-C-Ta-type alloys, the TaC-reinforcement principle in austenitic Fe-Ni base alloys, by elaborating and characterizing three alloys belonging to this system. The stable microstructures of these alloys were studied at 1000°C, 1100 and 1200°C, and the solidus and liquidus temperatures were determined. All these data can be used to evaluate and improve databases for thermodynamical calculations about alloys belonging to this alloy family.

In this work such a comparison was done between these experimental measurements and calculations performed using Thermo-Calc and a not complete database.

2. Experimental method

2.1. Elaboration of the alloys, metallographic characterization and thermal analysis measurements

All the studied alloys are chromia-formers and then hot corrosion-resistant since they contain near 30wt.% of chromium. They also contain tantalum, with two targeted contents: 3wt.% for the FENITA1 and FENITA2 alloys, and 6wt.% for the FENITA3 alloy. Thus, these alloys are similar to the FETA1, FETA2 and FETA3 ferritic iron-base alloys previously studied [5]. Carbon is present in the three alloys with a low content (0.2wt.%) in FENITA1 and a higher content (0.4wt.%) in FENITA2 and FENITA3. The synthesis of the alloys were achieved by mixing and fusion of pure elements (>99%, Alfa Aesar), using a high frequency induction furnace (CELES), under 300mbar of argon gas. They solidified in the water-cooled copper crucible of the furnace (leading to 100g compact ingots) and were heat-treated in a tubular resistive furnace to release the possible stresses due to solidification or to the following cooling down to room temperature. The cycle was: heating with 20°C/min, 2 hours stage at 1000°C and slow cooling in closed furnace. Several types of samples were cut for tests: approximately 2 x 2 x 8 mm³ for thermal analysis tests and 10 x 10 x 3 mm³ for high temperature exposures. Microstructures after solidification and stress-releasing heat treatment were investigated on samples embedded in cold resin (Araldite CY230 +Strengtheners Escil HY956) and polished with SiC paper from 120 to 1200 grid under water and finished with 1µm diamond pastes. A XL30 Philips Scanning Electron Microscope (SEM) was used for metallographic observations, with the Back Scattered Electrons mode (BSE) under an acceleration voltage of 20kV. The Energy Dispersion Spectrometry (EDS) device of the SEM allowed to analyze the obtained Fe, Ni, Cr and Ta contents to know the obtained global chemical composition of the whole alloy (except carbon which cannot be analyzed by EDS). This was done on five randomly selected areas, each of about 0.17mm², with calculation of the average value.

Solidus and liquidus temperatures were measured by Differential Thermal Analysis (DTA) using a Setaram TG-ATD apparatus. The thermal cycle was constituted by a first heating rate at 20 °C/min up to 1200 °C, a second heating rate at 5 °C/min from 1200 up to 1500 °C, a cooling at 5 °C/min from 1500 down to 1200°C and a second cooling at 20 °C/min down to room temperature. Beginnings and ends of fusion and solidification were measured and led to the solidus and liquidus temperatures. Two possible values of the solidus temperature (resp. liquidus) were considered: the beginning of fusion (resp. end) and the average value of the beginning of fusion (resp. end) and of the end of solidification (resp. beginning of solidification).

2.2. High temperature exposures, metallographic characterization and thermodynamic calculations

Three samples of each alloy were exposed for 50 hours to three different temperatures (1000, 1100 and 1200 °C), using a high temperature furnace with a heating rate of 20 °C/min and a cooling rate of 10 °C/min. The natures of the carbides seen on the metallographic samples were identified using Wavelength Dispersion Spectrometry (WDS) with a CAMECA SX100 microprobe. WDS measurements performed on five

different locations led to average values for the matrix composition. The microstructures were quantitatively characterized by taking five x500-pictures randomly selected on each sample using the SEM in BSE mode. The different phases, matrix (grey), chromium carbides (dark) and tantalum carbides (white), were separated from one another and their surface fractions measured using the Photoshop CS software of Adobe. Each surface fraction was the average of five measures, and was supposed to be close to the corresponding volume fractions. Fractions of carbides were also determined from the Cr and Ta contents in the whole alloy and in the matrix, following a procedure already described in the first article [5] which presents the equations that were used.

The Thermo-Calc software [6] and the database SSOL (SGTE) [7] were used to carry out calculations. The SSOL database already contained the descriptions of the binary and ternary systems Fe-Ni [8], Fe-C [9], Ni-C [10], Fe-Cr [11], Ni-Cr [12], C-Cr [13], Fe-C-Cr [14], Ni-C-Cr [15], Fe-Ni-C [10], Fe-Ni-Cr [16] and Fe-Ni-C-Cr [17]. The descriptions of other systems were added: Ni-Ta [18], Ta-C [19] and Ta-Cr [20]. Thermo-Calc calculations were performed for each alloy by considering the chemical composition determined by EDS measurements on the obtained alloys. The following quantities were calculated:

- the solidus temperature and the liquidus temperature,
- the chemical composition of matrix,
- the phases present at 1000 °C, 1100 °C and 1200 °C, and their mass fractions that were converted into volume fractions according to (1):

$$f_v[\phi_j] = \left(\frac{f_w[\phi_j]}{\rho_{\phi_j}} \right) / \sum_i \left(\frac{f_w[\phi_i]}{\rho_{\phi_i}} \right) \quad (1)$$

in which $f_v[\phi_j]$, $f_w[\phi_j]$ and ρ_{ϕ_j} are respectively the volume fraction, the weight fraction and the density of the phase ϕ_j . The densities (expressed in g.cm^{-3}) are 7.6 for matrix (as given in [21] for austenitic steels and verified by direct measure on samples), 6.941 for Cr_7C_3 , 6.953 for Cr_{23}C_6 and 14.5 for TaC [22].

3. Results

3.1. Initial microstructures and obtained chemical compositions

The initial microstructures of the three alloys can be seen in Fig. 1, Fig. 2 and Fig. 3, together with the microstructures of the same alloys after exposures to the three temperatures. Initially, the FENITA1 and FENITA3 alloys contained essentially interdendritic tantalum carbides, which obviously formed an eutectic with the matrix. There are carbides in double density for FENITA2 compared to FENITA1 which contains less carbon and tantalum. The FENITA2 alloy initially contains the same amount of TaC carbides as FENITA1, but also chromium carbides which are darker than the matrix.

Table 1 presents both the obtained chemical compositions of the three alloys measured by EDS (**in bold characters**) and the targeted ones for comparison. The nickel, chromium and tantalum contents were all well respected. Here too, it was not possible to obtain a sufficiently accurate value of the carbon content, but the targeted value can be considered well respected for the same reason as already given in [5].

3.2. Solidus and liquidus temperatures

Differential Thermal Analysis experiments led to heating and cooling curves on which temperatures of solidus and liquidus can be determined. Solidus temperatures were supposed to be either the temperature of beginning of fusion or the average value of the temperatures of beginning of fusion and of end of solidification, respectively for heating and cooling. Liquidus temperatures were taken either as the temperature of end of fusion or as the average temperature of end of fusion and of beginning of solidification. Their values are given in Table 2. The solidus temperatures of the two alloys exhibiting only TaC carbides (FENITA1 and FENITA3) are very close to one another, while the solidus temperature is sensibly lower for the third alloy in which chromium carbides are also present (FENITA2). The liquidus temperature is decreased by the presence of more carbon and/or more tantalum.

3.3. Microstructures after exposures to high temperature and surface fractions of carbides

Fig. 1, Fig. 2 and Fig. 3 respectively displays selected BSE images of the FENITA1, FENITA2 and FENITA3 alloys after exposure for 50 hours at 1000, 1100 and 1200°C, together with the corresponding initial microstructures just after solidification and stress-releasing heat treatment. The observed phases are on the one hand a Fe(Ni, Cr) austenitic matrix with a dendritic morphology, and on the other hand interdendritic carbides with an eutectic morphology which are either TaC tantalum carbides or Cr₂₃C₆ chromium carbides (according to WDS measurements).

For all temperatures the FENITA1 alloy contains essentially TaC carbides, with sometimes rare chromium carbides. There is qualitatively no real difference between the initial microstructure and the one after exposure at 1000°C. Solidification segregations are obviously smoothed by the high temperature exposures, and the surface fraction of tantalum carbides at the stable state decreases when temperature increases from 1000 to 1200°C.

The FENITA2 alloy contains both tantalum carbides and chromium carbides. Microstructures after high temperature exposures have slightly evolved from the initial microstructural state. Cr₂₃C₆ chromium carbides tend to become coarser or rounder when the exposure temperature increases, what is a frequent shape evolution that allows decreasing the carbide/matrix interface energy.

TaC remains the unique carbide existing in FENITA3 at all temperatures but they appear to be broken/rounder and their surface fraction is decreased when temperature is higher. These carbides are obviously affected by a fragmentation phenomenon that also results from a reduction of the interfacial carbide/matrix energy.

For each temperature five pictures were randomly taken per alloy with the MEB in BSE mode. The results of the image analysis performed on these pictures are given (**in bold characters**) in Table 3, which contains the average value and the interval of uncertainty (average value +/- standard deviation).

The FENITA1 alloy contains essentially TaC with a surface fraction of about 2.7% and which does not really depend on temperature. The chromium carbides must be

considered as trace carbides since their surface fraction is lower than 0.5% and decreases with temperature to totally disappear.

The TaC surface fractions in the FENITA2 alloy is of the same order of magnitude as the previous alloy. On the contrary the Cr_{23}C_6 carbides are significant since their surface fractions are higher than 1% (and it increases with temperature).

At high temperature the FENITA3 alloy only contains TaC carbides, with a surface fraction that can reach 10%.

3.4. Chemical compositions of the alloys matrix

The chemical compositions of the matrix are given (**in bold characters**) in Table 4 for all the alloys and for the three temperatures. Each content is the average value of 5 WDS spot analysis values obtained with microprobe. For all alloys the nickel and chromium contents in the matrix remain nearly constant when the exposure temperature varies. Tantalum is present in matrix in solid solution in all cases. Its content is comprised between 1 and 2wt.% and either increases with temperature (FENITA1 and FENITA3), or on the contrary decreases with temperature (FENITA2).

4. Comparisons with thermodynamic calculations

4.1. Computed solidus and liquidus temperatures

Thermodynamic calculations were all performed from the real chemical compositions given in Table 1. Some of these calculations led to the solidus and liquidus temperatures added in Table 2, for comparison with the experimental DTA measures.

The calculated solidus temperatures are all under 1300°C and seem to decrease when the carbon content becomes higher and to increase when the tantalum content is doubled. On the contrary, the calculated liquidus temperature decreases when the contents of the alloying elements become higher.

There is a significant mismatch between the calculated solidus temperature and the beginning of fusion, more for the FENITA1 and FENITA3 alloys than for the FENITA2 alloy the fusion of which begins with the melting of the eutectic chromium carbides. Differences between calculations and experiments are sensibly lower when the average values (between the start of fusion and the end of solidification) are considered. Similar observations can be made concerning the liquidus temperatures.

4.2. Computed matrix chemical compositions

Table 4 displays the chemical compositions of the matrix of all alloys obtained from Thermo-Calc calculations, together with the measured contents. The calculated Cr content slightly increases with temperature for FENITA1 and FENITA2, while it is the contrary for the FENITA3 alloy: this is almost not significant but this could be explained by an increasing of the volume fraction of matrix that was restricted at 1000°C because of the high TaC volume fraction. The calculated Ta content in the

matrix is significant for all alloys and all temperatures. It increases when the Ta content of the whole alloy is doubled (from FENITA1 and 2 to FENITA3) and when temperature increases. Carbon exists in matrix according to calculations, and its content increases with temperature.

There is a good correspondence between calculations and WDS measurements regarding chromium. On the contrary, some disagreements appear about tantalum and carbon. According to calculations Ta contents should be lower than measured on real samples, and carbon should be present in slightly higher quantities in the matrix than detected by microprobe.

4.3. Computed volume fractions of carbides

Figure 4 shows the positions of the three alloys in isothermal sections of the Fe-30wt.%Cr-xC-yTa phase diagram at T=1200°C, 1100°C and 1000°C and the mass fractions of carbides computed by Thermo-Calc are given in Table 3, as well as the corresponding volume fractions (*in italic characters*). The volume fractions of Cr₂₃C₆ and of TaC deduced from the chromium and tantalum contents of the matrix are added, under these calculated values and above the surface fractions measured by image analysis.

Calculations led to the existence of only TaC in the FENITA1 alloy at 1200°C while Cr₂₃C₆ would also exist in this alloy at 1100 and 1000°C. The fractions of the two types of carbides classically decrease with temperature. This is qualitatively of good agreement with the metallographic results but the calculated TaC volume fractions are sensibly lower than image analysis gave. There is a better correspondence when the volume fractions deduced from the chemical compositions of matrix and the whole alloy are considered. However calculated TaC volume fractions are always 50% higher than the values determined from the contents differences between alloy and its matrix.

According to calculations, both chromium carbides and tantalum carbides exist in the FENITA2 alloy, whatever the considered temperature, and their fractions at the stable state decrease when temperature increases.

The calculated volume fractions do not agree with the image analysis results since calculated chromium carbides represent volume fractions sensibly higher than the surface fractions measured by image analysis while it is the contrary for tantalum carbides, i.e. like for FENITA1. The volume fractions deduced from Cr and Ta contents differences between alloy and matrix show a better agreement with calculations, notably for 1200°C, but relative differences up to 50% are often observed.

For the FENITA3 alloy, calculations show that the TaC phase is the only type of carbide existing in the alloy at the three temperatures. The absence of chromium carbide is also found by image analysis while alloy/matrix Cr contents differences would show that these carbides may exist in the alloy. Concerning the tantalum carbides, the calculated TaC fractions are large but decrease with temperature, and image analysis led to values which are higher than calculated values. Here too the agreement is better with

TaC volume fractions deduced from the chemical compositions of the alloy and its matrix, than with fractions measured by image analysis.

5. Discussion

The presence of tantalum lead to high fractions of tantalum carbides in these austenitic iron/nickel base alloys, but less than for the ferritic iron base FETA alloys that were previously studied [5]. More precisely, it appears that the metallurgical behavior of these FENITA alloys is logically intermediate between the FETA alloys and the Ta-containing chromia-forming nickel base alloys studied in a previous work [4], concerning the relative quantities of chromium carbides and tantalum carbides as well as general instability of carbides (disappearance or growth of coarse acicular carbides). The same commentaries can also be made about the melting range. In order to facilitate the comparisons, the solidus and liquidus temperatures are summarized in Table 5 and Table 6, the Ta contents in matrix in Table 7 and the microstructural results in Table 8. In this last table, the volume fractions deduced from differences of Cr and Ta contents between alloy and its matrix were preferred. Indeed it obviously appears that the TaC phase, the white BSE colour of which is very useful for image analysis, unfortunately leads to overestimates of its surface fraction, for the same reasons as already given in the first article [5]. Indeed, here too the exposures to high temperature were long enough to reach the mass or volume fractions corresponding to the stable state. But the image analysis performed on BSE micrographs probably took into account not only the emerging carbides, but also a part of them that was just a few microns under the surface. It is also possible that surface and volume fractions are not always nearly equal. One can remark that such a problem did not really exist for the nickel-base alloys [4] since tantalum carbides represented very low surface fractions in these alloys.

When one observes what the solidus temperature becomes when the nickel is progressively replaced by iron as base element, it generally appears that the solidus temperature decreases (and reaches a minimal value for the FENITA alloy when chromium carbides are present). In addition, the presence of chromium carbides seems to maintain the solidus temperature at a low level in all cases. One can also see in the Fe-base system, that the solidus temperature seems to reach a minimal value for the intermediate 0.4%C-3%Ta composition, while it is true again in the Fe&Ni base system (but less pronounced) and it becomes false for the Ni-base system for which the solidus temperature remains almost constant. That seems to be a particularity of the Fe-base system that is progressively lost when iron is less and less present, to be replaced by the Ni-base system specific behavior (solidus temperature not dependent on C and Ta contents).

Concerning the liquidus temperature, similar commentaries can be made about the dependence of the liquidus temperature on the base elements: it sensibly decreases between the Fe base alloys and the (Fe,Ni) base alloys, but it remains at the same order of magnitude between the (Fe,Ni) base alloys and the Ni base alloys. In addition it can be noted that liquidus temperatures are particularly high for the Fe-base alloys. When the alloys become enriched in C and/or Ta, the liquidus temperature decreases for the Ni-base and (Fe, Ni) base systems while, on the contrary, it seems increasing for the Fe-base system (but only the Thermo-Calc values are available for FETA liquidus temperatures).

The tantalum weight contents in matrix are remembered in Table 7, in which it is clearly visible that the BCC iron matrix does not contain any significant amount of tantalum (less than 0.1% for all alloys and all temperatures). The Ta content is higher in the FCC (Fe, Ni) matrix (mainly between 1 and 2%) and even higher for the FCC nickel matrix (from 2 up to 4.2). On the iron side, it is clear that a main part of tantalum must form carbides while, on the nickel side, it can stay in solid solution in matrix. This leads to the observations resumed in the following table. Indeed one can see in Table 8 the volume fraction increasing for chromium carbides together with the volume fraction decreasing for tantalum carbides when one progressively quit the Fe-base system to approach the Ni-base system. This is true for all the temperatures and for all the carbon and tantalum contents of the alloy. It clearly appears that an iron base is more favorable to the TaC phase and less to the Cr₂₃C₆ phase than a nickel base.

6. Conclusions

The austenitic iron-nickel base alloys are able to contain significant volume fractions of tantalum carbides, however less than it was obtained in the iron-base alloys with similar carbon and tantalum contents. Compared to these ones, the TaC-strengthened Fe-Ni base alloys seem to be more metallurgically stable at high temperature, even if they are also less refractory. Less expensive than other superalloys, this type of alloys probably brings a better mechanical resistance at high temperature than tantalum-containing carbides-strengthened nickel base alloys. As it was also seen on TaC-reinforced iron-base alloys in the first article, the observation of the TaC phase with a SEM in BSE mode can lead to overestimation of the surface or volume fractions of tantalum carbides, qualitatively or quantitatively when image analysis is practiced. This can cause worse mismatches between thermodynamic calculations and surface fractions measurements, then deductions of volume fractions from the chemical composition differences between alloy and matrix must be preferred. Mismatches exist between calculated results and experimental results issued from measures on real samples. Then progress remains to be achieved, notably by enrichment of the used database with the descriptions of lacking systems. In this field the experimental results presented in this study can be of help to improve the databases concerning this Fe-Ni-Cr-C-Ta system, e.g. following the way already explained in the conclusions of the first article concerning the Fe-Cr-C-Ta system.

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TABLES

Table 1
Chemical compositions of the three studied alloys (in weight percent)
(average values of EDS measurements on six x250 areas)

Alloy	Chem. Comp.	Fe	Ni	Cr	Ta	C
FENITA1	targeted	bal.	33.4	30	3	0.2
	obtained	bal.	34.2 +/- 0.5	29.6 +/- 0.3	2.9 +/- 0.3	Supposed to be 0.2
FENITA2	targeted	bal.	33.4	30	3	0.4
	obtained	bal.	34.0 +/- 0.4	30.0 +/- 0.3	2.8 +/- 0.4	Supposed to be 0.4
FENITA3	targeted	bal.	33.4	30	6	0.4
	obtained	bal.	32.5 +/- 0.2	29.5 +/- 0.3	6.4 +/- 0.3	Supposed to be 0.4

Table 2
Solidus and liquidus temperatures of the three alloys measured by Differential Thermal Analysis (**in bold characters**) and computed by ThermoCalc

Alloy	Solidus Temp. (°C)	Liquidus Temp. (°C)
	DTA: heating only (average heating/cooling) Thermo-Calc	
FENITA1	1317 (1306)	1394 (1382)
	1287	1373
FENITA2	1292 (1282)	1394 (1367)
	1280	1359
FENITA3	1316 (1302)	1377 (1356)
	1292	1336

Table 3

Surface fractions of carbides measured using image analysis performed on SEM-BSE pictures for each alloy and each temperature (**in bold characters**); comparison with volume fractions deduced from the Cr and Ta contents of the matrix (normal characters) and with volume fractions calculated using Thermo-Calc (*italic characters*)

Alloy	<i>calculated volume fractions (%)</i> , vol. frac. from %Cr and %Ta in matrix (%) and surface fractions (%)	$M_{23}C_6$	TaC
FENI-1200°C	(calculated mass fractions) <i>calculated volume fractions</i>	0 <i>0</i>	2.46 <i>1.36</i>
	vol. frac. deduced from matrix %Cr and %Ta	0	0.86
	measured by image analysis	0	2.64 ±0.24
-TA1-1100°C	(calculated mass fractions) <i>calculated volume fractions</i>	0.27 <i>0.31</i>	2.59 <i>1.43</i>
	vol. frac. deduced from matrix %Cr and %Ta	0	0.94
	measured by image analysis	0	2.70 ±0.33
-TA1-1000°C	(calculated mass fractions) <i>calculated volume fractions</i>	0.40 <i>0.46</i>	2.69 <i>1.48</i>
	vol. frac. deduced from matrix %Cr and %Ta	0	1.00
	measured by image analysis	0.30 ±0.11	2.66 ±0.56
-TA2-1200°C	(calculated mass fractions) <i>calculated volume fractions</i>	3.55 <i>4.07</i>	2.45 <i>1.34</i>
	vol. frac. deduced from matrix %Cr and %Ta	5.17	1.39
	measured by image analysis	2.01 ±0.51	2.37 ±0.20
-TA2-1100°C	(calculated mass fractions) <i>calculated volume fractions</i>	3.83 <i>4.39</i>	2.56 <i>1.41</i>
	vol. frac. deduced from matrix %Cr and %Ta	3.11	1.12
	measured by image analysis	0.81 ±0.18	2.79 ±0.16
-TA2-1000°C	(calculated mass fractions) <i>calculated volume fractions</i>	3.97 <i>4.55</i>	2.66 <i>1.46</i>
	vol. frac. deduced from matrix %Cr and %Ta	2.42	1.03
	measured by image analysis	1.05 ±0.18	2.24 ±0.15
-TA3-1200°C	(calculated mass fractions) <i>calculated volume fractions</i>	0 <i>0</i>	6.03 <i>3.38</i>
	vol. frac. deduced from matrix %Cr and %Ta	0.94	2.69
	measured by image analysis	0	10.14 ±0.72
-TA3-1100°C	(calculated mass fractions) <i>calculated volume fractions</i>	0 <i>0</i>	6.25 <i>3.50</i>
	vol. frac. deduced from matrix %Cr and %Ta	2.21	3.03
	measured by image analysis	0	6.48 ±0.30
-TA3-1000°C	(calculated mass fractions) <i>calculated volume fractions</i>	0 <i>0</i>	6.39 <i>3.59</i>
	vol. frac. deduced from matrix %Cr and %Ta	0.81	3.00
	measured by image analysis	0.10 ±0.06	8.92 ±3.91

Table 4
Matrix chemical compositions at 1000°C, 1100°C and 1200°C,
analysed with WDS microprobe (**in bold characters**) and computed with Thermo-Calc

Alloy	Fe+Ni	Cr	Ta	C
		Weight contents analysed by microprobe (%) Calculated weight contents (%)		
FENITA1				
1200°C	bal.	31.6 +/- 0.2 30.3	1.43 +/- 0.04 0.56	0 0.05
1100°C	bal.	31.4 +/- 0.3 30.2	1.30 +/- 0.09 0.44	0 0.03
1000°C	bal.	31.4 +/- 0.1 30.2	1.20 +/- 0.06 0.34	0 0.01
FENITA2				
1200°C	bal.	27.7 +/- 0.9 29.0	0.46 +/- 0.03 0.55	0 0.05
1100°C	bal.	28.8 +/- 0.3 28.8	0.94 +/- 0.10 0.44	0.09 +/- 0.12 0.03
1000°C	bal.	29.2 +/- 0.3 28.6	1.09 +/- 0.35 0.34	0.31 +/- 0.33 0.01
FENITA3				
1200°C	bal.	30.5 +/- 0.2 31.4	1.92 +/- 0.09 0.80	0.05 +/- 0.07 0.03
1100°C	bal.	29.9 +/- 0.45 31.5	1.33 +/- 0.29 0.59	0.18 +/- 0.21 0.02
1000°C	bal.	30.7 +/- 0.1 31.6	1.36 +/- 0.06 0.44	0 0

Table 5
Calculated (**in bold**) and measured solidus temperatures (in Celsius degrees °C) of
tantalum-containing
chromia-forming alloys based on iron and/or nickel

Alloy types	Fe base*	Fe&Ni base**	Ni base***
“30Cr-0.2C-3Ta”	1419 (1408) 1436	1317 (1306) 1287	1310 (1298) 1303
“30Cr-0.4C-3Ta”	1320 (1302) 1298	1292 (1282) 1280	1315 (1301) 1299
“30Cr-0.4C-6Ta”	1405 (1390) 1471	1316 (1302) 1292	1304 (1302) 1280

*: ref [5] **: this study ***: ref [4]

Table 6
Calculated (**in bold**) and measured liquidus temperatures (in Celsius degrees °C) of
tantalum-containing
chromia-forming alloys based on iron and/or nickel

Alloy types	Fe base*	Fe&Ni base**	Ni base***
“30Cr-0.2C-3Ta”	>1500°C (>1500°C) 1525	1394 (1382) 1373	1409 (1399) 1377
“30Cr-0.4C-3Ta”	>1500°C (>1500°C) 1563	1394 (1367) 1359	1381 (1377) 1360
“30Cr-0.4C-6Ta”	>1500°C (>1500°C) 1713	1377 (1356) 1336	1377 (1374) 1346

*: ref [5] **: this study ***: ref [4]

Table 7
Tantalum weight contents in matrix for all alloys and all exposure temperatures
(in wt. %)

WDS wt.%Ta in matrix	Fe base*	Fe&Ni base**	Ni base***
“30Cr-0.2C-3Ta” at 1200°C	0.05	1.43	2.04
“30Cr-0.2C-3Ta” at 1100°C	0.06	1.30	2.39
“30Cr-0.2C-3Ta” at 1000°C	0.04	1.20	2.38
“30Cr-0.4C-3Ta” at 1200°C	0.02	0.46	2.78
“30Cr-0.4C-3Ta” at 1100°C	0.05	0.94	2.70
“30Cr-0.4C-3Ta” at 1000°C	0.05	1.09	2.40
“30Cr-0.4C-6Ta”- type at 1200°C	0.07	1.92	2.99
“30Cr-0.4C-6Ta” at 1100°C	0.07	1.33	4.22
“30Cr-0.4C-6Ta” at 1000°C	0.06	1.36	3.57

*: ref [5] **: this study ***: ref [4]

Table 8
Volume fractions of carbides (in %) deduced from the differences of chemical
compositions
between each alloy and its matrix, for the three temperatures

vol. %	Fe base*		Fe&Ni base**		Ni base***	
	Cr₂₃C₆	TaC	Cr₂₃C₆	TaC	Cr₂₃C₆	TaC
“30Cr-0.2C-3Ta” at 1200°C	0	1.12	0	0.86	3.07	0.03
“30Cr-0.2C-3Ta” at 1100°C	0	1.16	0	0.94	5.17	0
“30Cr-0.2C-3Ta” at 1000°C	0.30	1.17	0	1.00	3.18	0
“30Cr-0.4C-3Ta” at 1200°C	1.26	1.32	5.17	1.39	7.33	0.03
“30Cr-0.4C-3Ta” at 1100°C	2.08	1.33	3.11	1.12	9.94	0.11
“30Cr-0.4C-3Ta” at 1000°C	2.57	1.34	2.42	1.03	8.69	0.28
“30Cr-0.4C-6Ta” at 1200°C	0.00	3.32	0.94	2.69	4.94	1.72
“30Cr-0.4C-6Ta” at 1100°C	0.00	3.39	2.21	3.03	7.19	0.99
“30Cr-0.4C-6Ta” at 1000°C	0.04	3.44	0.81	3.00	6.92	1.40

FIGURES

Fig. 1. Initial microstructure of FENITA1 and microstructures after 50 hours-exposures at 1000°C, 1100°C and 1200°C (all pictures taken with MEB in BSE mode at x500)

Fig. 2. Initial microstructure of FENITA2 and microstructures after 50 hours-exposures at 1000°C, 1100°C and 1200°C (all pictures taken with MEB in BSE mode at x500)

Fig. 3. Initial microstructure of FENITA3 and microstructures after 50 hours-exposures at 1000°C, 1100°C and 1200°C (all pictures taken with MEB in BSE mode at x500)

Fig. 4. 1200°C, 1100°C and 1000°C sections of the Fe-33.4wt.%Ni-30wt.%Cr-xC-yTa phase diagram