Microstructures and hardness of model niobium-based chromium-rich cast alloys

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Abstract. Niobium is a candidate base for new alloys devoted to applications at especially elevated temperatures. Elaborating and shaping niobium-based alloys by conventional foundry may lead to mechanically interesting microstructures. In this work a series of charges constituted of pure elements were subjected to high frequency induction melting in cold crucible to try obtaining cast highly refractory Nb-xCr and Nb-xCr-0.4 wt.%C alloys (x=27, 34 and 37 wt.%). Melting and solidification were successfully achieved. The as-cast microstructures of the obtained alloys were characterized by electron microscopy and X-ray diffraction and their hardness were specified by Vickers macro-indentation. The obtained as-cast microstructures are composed of a body centered cubic (bcc) niobium dendritic matrix and of an interdendritic eutectic compound involving the bcc Nb phase and a $NbCr_2$ Laves phase. The obtained alloys are hard to cut and particularly brittle at room temperature. Hardness is of a high level (higher than 600Hv) and is directly driven by the chromium content or the amount of {bcc Nb $-$ $NbCr_2$} eutectic compound. Adding 0.4 wt.% of carbon did not lead to carbides but tends to increase hardness.

Keywords: niobium-based alloys; chromium; carbon; foundry; as-cast microstructures; hardness

1. Introduction

The hottest parts of the turbines used for transportation or for power generation tend working at temperatures which constantly increase. The used metallic alloys must be more and more refractory and more and more resistant against simultaneous thermal, mechanical and chemical solicitations. To go beyond the actual alloy performances (insured for example by the single crystalline nickel-based γ/γ' alloys (Sims and Hagel 1972, Bradley 1988, Donachie and Donachie 2002, Nembach and Neite 1985), many researches are focused on the strengthening of cobalt alloys by γ' precipitation (Cui *et al.* 2006, Mishra *et al.* 2013), on Co-based alloys rich in heavy elements (Klauke *et al.* 2009, Gorr *et al.* 2012), on high entropy alloys containing very refractory elements (Gorr *et al.* 2016), for instance. Other ways may be also considered such as alloys based on elements more refractory than nickel, cobalt and iron, but taking the same reinforcing principles

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as recent developments concerning these later alloys, for example by associating refractory metallic bases with carbides highly stable at high temperature. Such carbides are for instance MC carbides in which M is tantalum, hafnium, zirconium… and C is carbon (e.g. of the MC type (Berthod 2009)). When elaborated by conventional foundry the obtained MC carbides are of a eutectic nature (imbricated with matrix), script-like shaped and located in the interdendritic spaces. Such principles led to promising high temperature mechanical properties, bringing to equiaxed polycrystalline alloys a new interest for applications at elevated temperatures. More recently, chromium-based alloys, consisting of a body centered cubic Ni-saturated matrix mixed with a face centered cubic nickel Cr-saturated phase, were synthesized by foundry (Conrath and Berthod 2016). This demonstrated the possibility of elaboration by casting of alloys based on this rather refractory element (Cr, 1870°C). Niobium is another metallic element candidate to play the role of the base of highly refractory cast alloys. With its melting point which is a little higher than 2400°C (Shaffer 1964), niobium is, together with Mo, intermediate between the (Ni, Co, Fe, Cr) group and more refractory elements such as Ta (near 3000°C) or W (near 3500°C). Used as base element it may lead to alloys which are highly mechanically resistant at elevated temperature (Wojcik 1998), working temperatures however limited to 1300°C (Perepezko 2009). Some Nb-based alloys may contain carbides when carbon is added to the composition (Ding and Jones 2008). The behavior in oxidation at high temperature may be critical (Gebhardt *et al.* 1972) and the presence of Si or Al, less commonly Cr, are required to avoid catastrophic behavior for the bulk materials (Kofstad 1988, Young 2008). Producing Nb-based alloys maybe done by powder metallurgy but also by melting-solidification: arc-melting (Sha *et al.* 2004) or directional solidification (Guan *et al.* 2004), for instance.

The present work concerns cast niobium-based alloys alloyed with high quantities of chromium. The chosen Cr contents aim to allow these alloys resisting oxidation by gases as well as corrosion by molten substances such as sulphides mixtures, CMAS (mixture of CaO, MgO, Al_2O_3 and SiO_2) or glasses… Before proceeding to the elaboration of niobium-based alloys with complex chemical compositions, one preliminarily wished to elaborate model alloys using an apparatus which recently allowed elaborating equiaxed chromium-based alloys by casting. Despite probable higher difficulties than the ones already encountered for chromium alloys, one can hope melting and shaping such niobium based alloys and explore the microstructures of simple alloys destined to be the bases of more complex future ones.

2. Experimental details

2.1 Selection of the alloys to study

The present work was centered on binary and ternary alloys, based on niobium and containing several amounts of chromium. Since niobium is known as being easy to oxidize, the chromium contents were chosen by taking as reference the Cr-contents in the cobalt-based alloys which are less resistant in high temperature oxidation than the Ni-based superalloys. The chromium contents which were then chosen are 27 wt.% Cr ("low Cr", a little lower than the limit which allows cobalt-based alloys being chromia-forming in many situations), 34 wt.% Cr ("medium Cr", high enough for many cobalt-based alloys) and 37 wt.% ("high Cr", high but not exaggerated). In order to start observing the reaction of these bases in presence of carbon, before adding strong carbide-former element as fourth element, in future works), three other alloys were also considered. These

ones contain the same chromium quantities, but also 0.4 wt.% C, carbon content which can be considered as representative of the C contents generally found in carbide-strengthened nickel-based or cobalt-based superalloys. Thus, there are six different cast Nb-Cr-C alloys which were subjected to investigations here: three binary compositions (in weight percent), Nb-27Cr, Nb-34Cr and Nb-37Cr and three ternary ones (wt.% too), Nb-27Cr-0.4C, Nb-34Cr-0.4C and Nb-37Cr-0.4C.

2.2 Casting of the chosen alloys

The alloys were elaborated using a high frequency induction furnace (CELES, France). The masses of pure elements (Alfa Aesar, purity > 99.9 wt.%), prepared to obtain ingots weighing about 10g, were placed in the metallic crucible (copper) cooled by a continuous water circulation. A silica tube was then placed to isolate the melting chamber from air laboratory. Primary vacuum was realized in this chamber, until reaching about 5×10^{-2} mbars inside. Pure argon was thereafter injected until reaching 800 mbars. Vacuum was realized again, followed by argon injection. This cycle {pumping + argon injection} was done at least three times, before obtaining a 300 mbars Argon atmosphere in the fusion chamber.

The internal atmosphere being now inert (not oxidant), heating was then performed. The applied voltage was increased from 0 to 2500 V (frequency: about 110 kHz), with a first {30 seconds}-stage to allow thermal homogenization of the still solid parts of pure niobium and pure chromium, as well as pure graphite in the case of the ternary alloys. New increase in power until 5 kV was then achieved. Power was maintained at this value for 5 minutes, which allowed the obtained liquid alloy to become homogenized. Cooling was carried out by decreasing progressively voltage down to zero. After a {30 minutes}-cooling of the solidified alloy a new whole thermal cycle was applied to be sure to have totally melted and chemically homogenized the alloy. Some preliminary tests were carried out for validating this protocol. This one was thereafter systematically followed for all elaborations. Applying it allowed avoiding any risk of possible presence of inclusions of not melted small Cr or Nb parts in the ingots and obtaining chemically homogeneous alloys.

2.3 Cutting, sample preparation and characterization

The obtained ingots were cut using an Abrasimet Delta metallographic cutter (Buhler). A part from each ingot was embedded in a cold resin mixture (ESCIL, France), then ground. Grinding using SiC papers from 120-grit up to 1200-grit, was followed by ultrasonic washing in water to remove any SiC hard particles. Thereafter, the samples were polished with textile disk enriched with 1µm alumina (Al_2O_3) particles.

The embedded and polished samples were subjected to X-ray diffraction (Philips X'Pert Pro, Cu Kα, λ=1.5406Å) to identify the phases present in the alloys and to metallographic observations to discover the obtained microstructures and analyze the chemical compositions. A Scanning Electron Microscope (SEM, JEOL JSM 6010 LA), equipped with an Energy Dispersion Spectrometer, was used in Back Scattered Electrons mode (BSE) to visualize the as-cast microstructures and to control the global (full frame) and local (spot) compositions of the whole alloy and the different phases. Electronic micrographs were taken in BSE mode and analyzed using the image analysis tool of the Photoshop CS software (Adobe) to specify the surface fractions of the different phases.

Indentation tests were performed to measure the global hardness of the obtained alloys. This
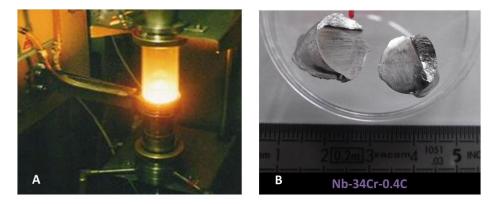
Fig. 1 Photograph of the hot part of the High Frequency Induction furnace, when melting one of the alloys (A); one of the obtained ingots after cutting, illustrating the brittle final rupture occurred near the end of cutting (B)

was done using a Testwell Wolpert machine. Tests were carried out by following the Vickers method, with a {10 kg} load. About ten indentations were performed by ingot. Some of the marks were thereafter examined using the SEM, in Secondary Electrons (SE) mode.

3. Results

3.1 Observation with the naked eye

During the elaboration (illustration in Fig. 1-A) carried out following to the protocol described above, the thermal cycle obviously led to a total melting of the mix of pure elements and a good homogenization, at least after the second solidification. Notably, no not-melted part was observed inside the ingots after their cutting in two halves (illustration in Fig. 1-B). Cutting was very hard to do, because an apparent high hardness of the castings. For all alloys, the ingot suddenly broke at cutting end. The breaking fracture surface clearly evidenced a brittle rupture (Fig. 1-B).

3.2 Microstructures of the obtained alloys

The as-cast microstructures of the binary and ternary alloys are illustrated in Fig. 2 by SEM micrographs taken in the BSE mode. One can see that the alloys are all double-phased, with the presence of white dendrites and grey interdendritic areas. Whatever the type of alloy (binary Nb-Cr or ternary Nb-Cr-C), the higher the chromium content, the lower the apparent surface fraction of the Nb-based phase. The as-cast microstructures of the ternary alloys are very similar to the ones of the binary alloys containing the same chromium content (example in Fig. 2, comparison of B and B′). Notably no carbides were observed in the carbon-containing alloys. For all alloys, EDS spot analysis showed that the white phase is a Nb-based one containing 14 to 16 wt.% Cr and the grey constituent is richer in Cr than the former (about 40 wt.%Cr).

Observed at higher magnification (Fig. 3) it appears that the grey constituent is, in fact, a finely structured mix of white phase and dark phase. As demonstrated by some EDS spot analyses performed in locations where these constituents were coarse enough, the white phase is the same
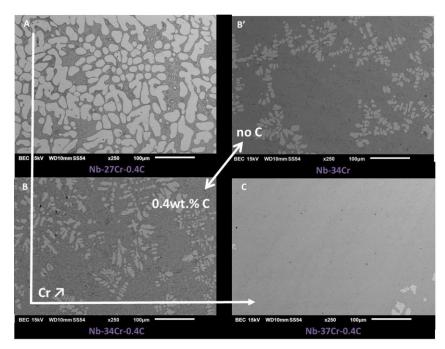
Fig. 2 SEM/BSE micrographs of some obtained as-cast microstructures, illustrating the evolution when the chromium content increases (A, B, C) and the absence of microstructure difference when carbon is present (B′, to compare to B); general view (×250 magnification)
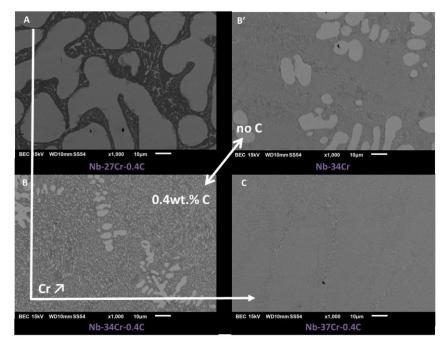

Fig. 3 SEM/BSE micrographs of some obtained as-cast microstructures, illustrating the evolution when the chromium content increases (A, B, C) and the absence of microstructure difference when carbon is present (B′, to compare to B); detailed view (×1000 magnification)
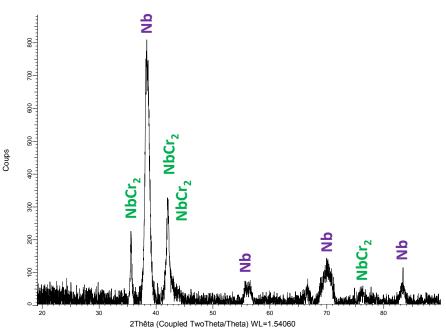
Fig. 4 XRD diffractogram obtained on the as-cast Nb-34Cr alloy, allowing the identification of its two constituting phases

as the one constituting the dendrites (about 15 wt.% Cr) and the dark phase is much richer in chromium (about 50 wt.% Cr). X-ray diffraction runs allowed completing the identification of the two existing phases: one is a body centered cubic Nb-based solid solution and the other is a $NbCr_2$ Laves phase (Fig. 4).

Image analyses performed in several locations (SEM/BSE, magnification rated to ×1000) allowed measuring the corresponding surface fractions. The results are plotted versus the chromium content for the ternary alloys in Fig. 5. The results obtained for the binary ones are totally similar for a given chromium content in alloy. Only the results obtained for the low chromium alloys (high surface fraction of dendrites) can be considered as good. Indeed the fineness of the grey constituent induces severe loss of accuracy for the determination of the white phase. Therefore one must consider the surface fraction plotted versus the chromium content in Fig. 5 as being the surface fraction of Nb-phase dendrites only.

3.3 Vickers indentation results

The indentations, performed following the Vickers method with a load of 10 kg, evidenced very high level of hardness. Some values are first given together with the SEM/SE micrographs of the corresponding marks in Fig. 6 for the binary alloys and Fig. 7 for the ternary alloys. All these values are high and it appears that hardness seems first increasing when the chromium content increases and second hardness is higher when 0.4 wt.% of carbon is present. These two tendencies are confirmed when all the results are taken into account (Fig. 8). When one carefully observes the marks one can notice cracks developing from each edge of the mark over distances of the same level of magnitude as the marks dimensions. These cracks are parallel to the diagonals of the

marks and seem having propagated preferentially through the grey constituent.

4. Discussion

The used high frequency induction furnace and the followed protocol obviously, succeeded in obtaining these highly refractory Nb-based alloys. No unmelted parts of initial pure niobium or pure chromium were noticed and the chemical compositions were respected.
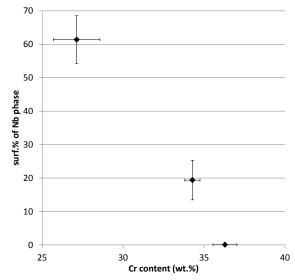

Fig. 5 Surface fraction of the BCC Nb-based solid solution (more precisely dendritic part) plotted versus the chromium content (average and standard deviation of five local values issued from EDS {×250 full frame} analyses and from five {×1000 BSE pictures} image analysis results)
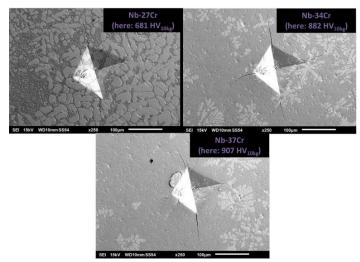

Fig. 6 SEM/SE micrographs of Vickers indentation marks (load: 10 kg) and of the corresponding hardness values (here: the three binary Nb-Cr alloys)
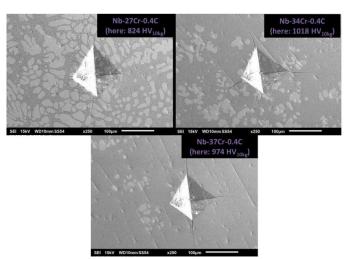
Fig. 7 SEM/SE micrographs of Vickers indentation marks (load: 10 kg) and of the corresponding hardness values (here: the three ternary Nb-Cr-C alloys)
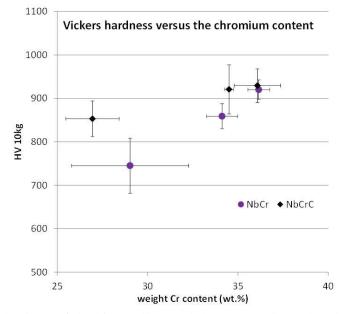

Fig. 8 The Vickers hardness of the binary alloys and the ternary alloys plotted versus the chromium content; influence of the presence of carbon

Concerning the binary alloys, the identified phases and constituent are in good agreement with the Nb-Cr diagram reminded in (Thoma *et al.* 1997) and presented in Fig. 9 where it is enriched with the positions of these three alloys.

Indeed, the obtained microstructures are composed of a coarse Nb-based phase (obviously crystallized during the pre-eutectic part of solidification) and of a finely structured constituent composed of Nb-phase and of $NbCr_2$ phase (Laves phase). The chemical compositions of these two phases (pre-eutectic and eutectic Nb-phase, eutectic Laves phase), respectively 15 wt.% Cr
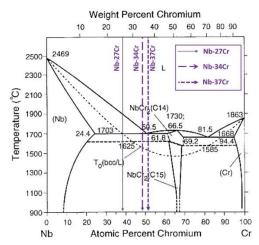
Fig. 9 Position of the three binary alloys in the Nb-Cr diagram

Table 1 Theoretic microstructures for the three binary alloys at several temperatures
("ε": arbitrary very low quantity, much less than 1 mss.%, "mss.%": mass fraction of phase, "BCC Nb": the body centered cubic Nb-based solid solution, "ctg.": containing)

Temperature (°C)	Nb-27Cr alloy (hypo-eutectic)	Nb-34Cr alloy (hypo-eutectic)	Nb-37Cr alloy (almost eutectic)
≈ 1900°C	(100-ε) mss.% of Liquid (ctg. 27 wt.%Cr) + ε mss.% of BCC Nb (ctg. 11 wt.%Cr)	100% LIQUID	100% LIQUID
≈ 1740°C	↓	(100-ε) mss.% of Liquid (ctg. 27 wt.%Cr) + ε mss.% of BCC Nb (ctg. 11 wt.%Cr)	↓
≥ 1700°C	55.6 mss.% of Liquid (ctg. 36.4 wt.%Cr) + 44.4 mss.% of BCC Nb (ctg. 15.3 wt.%Cr)	88.8 mss.% of Liquid (ctg. 36.4 wt.%Cr) + 11.2 mss.% of BCC Nb (ctg. 15.3 wt.%Cr)	100% LIQUID
≤ 1700°C	44.4 mss.% of primary BCC Nb (ctg. 15.3 wt.%Cr) + 55.6 mss.% of eutectic composed of 34.7 mss.% eut. BCC Nb & 65.3 mss.% of NbCr$_2$ (i.e. 63.7 mss.% BCC Nb and 36.3mss.% NbCr$_2$)	11.2 mss.% of primary BCC Nb (ctg. 15.3 wt.%Cr) + 88.8 mss.% of eutectic composed of 34.7 mss. eut. BCC Nb & 65.3 mss.% of NbCr$_2$ (i.e. 42.0 mss.% BCC Nb and 58.0 mss.% NbCr$_2$)	100 mss.% of eutectic composed of 34.7 mss.% eut. BCC Nb (ctg. 15.3 wt.%Cr) & 65.3 mss.% of NbCr$_2$
1000°C	47.1 mss.% of BCC Nb (ctg. 4.64 wt.%Cr) + 52.9 mss.% of NbCr$_2$ (ctg. 52.1 wt.%Cr)	38.1 mss.% of BCC Nb (ctg. 4.64 wt.%Cr) + 61.9 mss.% of NbCr$_2$ (ctg. 52.1 wt.%Cr)	31.8 mss.% of BCC Nb (ctg. 4.64 wt.%Cr) + 68.2 mss.% of NbCr$_2$ (ctg. 52.1 wt.%Cr)

*: calculated using the values of density at 20°C of BCC Nb (8.57 g/cm3) and of NbCr2 (7.66 g/cm3)

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Table 2 Theoretic values of the volume fractions of the two phases just after solidification and at 1000°C (issued from the mass fractions presented in Table 1)

Temperature (°C)	Nb-27Cr alloy (hypo-eutectic)	Nb-34Cr alloy (hypo-eutectic)	Nb-37Cr alloy (almost eutectic)
Just after solidification	Mass fractions: 63.7 mss.% of BCC Nb (ctg. 15.3 wt.%Cr) and 36.3 mss.% of NbCr$_2$ (ctg. 47.5 wt.%Cr)	Mass fractions: 42.0 mss.% of BCC Nb (ctg. 15.3 wt.%Cr) and 58.0 mss.% of NbCr$_2$ (ctg. 47.5 wt.%Cr)	Mass fractions: 34.7 mss.% of BCC Nb (ctg. 15.3 wt.%Cr) and 65.3 mss.% of NbCr$_2$ (ctg. 47.5 wt.%Cr)
	Corresponding volume fractions*: 61.1 vol.% BCC Nb 38.9 vol.% NbCr$_2$	Corresponding volume fractions*: 39.3 vol.% BCC Nb 60.7 vol.% NbCr$_2$	Corresponding volume fractions*: 32.2 vol.% BCC Nb 67.8 vol.% NbCr$_2$
1000°C	Mass fractions: 47.1 mss.% of BCC Nb (ctg. 4.64 wt.%Cr) and 52.9 mss.% of NbCr$_2$ (ctg. 52.1 wt.%Cr)	Mass fractions: 38.1 mss.% of BCC Nb (ctg. 4.64 wt.%Cr) and 61.9 mss.% of NbCr$_2$ (ctg. 52.1 wt.%Cr)	Mass fractions: 31.8 mss.% of BCC Nb (ctg. 4.64 wt.%Cr) and 68.2 mss.% of NbCr$_2$ (ctg. 52.1 wt.%Cr)
	Corresponding volume fractions*: 44.3 vol.% BCC Nb 55.7 vol.% NbCr$_2$	Corresponding volume fractions*: 35.5 vol.% BCC Nb 64.5 vol.% NbCr$_2$	Corresponding volume fractions*: 29.4 vol.% BCC Nb 70.6 vol.% NbCr$_2$

and 50 wt.% Cr are close to the ones at the eutectic temperature. This suggests that the cooling was too fast to allow chromium diffusion and then to allow the phases' chemical compositions and mass fractions to follow the thermodynamic equilibrium requirements. The theoretic mass fractions of the two phases calculated from the binary diagram and listed in Table 1 are converted in volume fractions in Table 2. They confirm the better correspondence with the metallurgical state at the eutectic temperature than with lower temperatures. Anyway, the observed microstructures of the as-cast binary alloys may be considered as being in good agreement with the binary diagram since the differences are probably due to kinetic reasons. Concerning the as-cast ternary alloys, which were remarkably similar to the binary ones for the same chromium contents, no carbides have neither crystallized during solidification, nor precipitated during the solid state cooling. This remains to be compared to ternary diagrams if available, or to thermodynamic calculations using adequate software and database. The carbon atoms seem having been trapped in the Nb-based phase or in the Laves phase during solidification. These carbon atoms may influence the hardness of these phases. Since the hardness difference between the binary alloys and the ternary alloys increased when the chromium content decreased and thus when the fraction of Nb-phase increases, one guess that carbon is present in solid solution in the Nb-phase rather than in the Laves phase.

The characterization of the as-cast microstructures also informed us about the morphologies of these phases. Notably, the dendritic morphology of the Nb phase can be favorable to the mechanical properties at high temperature. The Laves phase, which is obviously much harder than the Nb-phase, as demonstrated by the increase in hardness when the surface fraction of the Nb phase decreases, seems mechanically favorable too. One can remind here that Laves phases of various types (Cr$_2$Ta for example) are sometimes welcome in refractory alloys to which they bring

mechanical resistance at high temperature. Unfortunately, the Laves phase may also induce brittleness at high temperature as well as at room temperature. This was observed here by noticing the crack propagation from the indentation marks.

5. Conclusions

Niobium and chromium are two metallic elements particularly refractory. Their very high melting points allow them constituting alloys with high values of solidus temperatures, condition compulsory (but not sufficient) to achieve high mechanical strength at elevated temperatures of use.

Despite the high refractoriness of Nb and Cr, the preparation by casting of alloys involving these elements was successful here. Indeed, defects-free homogenous alloys were obtained, notably without any remaining unmelted parts. However, this good result, obtained here with ingots of only several tens grams it is true, risks to be more difficult to reproduce for elaboration of much higher alloy volumes, typical of many real industrial pieces. It is possible that it will be compulsory to use mother alloys (involving foreign elements such as iron for example) to favor the success of the fabrication by high frequency induction foundry of components weighing several kilograms.

In the microstructure field, the alloys obtained in this work contain constituents with mechanical properties and morphologies of great interest for high temperatures applications involving stresses. The two phases existing in the microstructures lead to a particularly high hardness of the alloys at room temperature. This lets expecting high strength at high temperature. The dendritic morphology of one of the two phases, typical solidification morphology which is achievable by foundry way only, represents an important asset for a good mechanical behavior at elevated temperatures, as demonstrated by many more classical superalloys. Unfortunately, property which often accompanies high level of hardness, these first six alloys are too brittle at room temperature and they probably present also low toughness at higher temperatures. Consequently, one can fear high difficulties of machining and low resistance to thermal cycling. Thus, in parallel with the optimization of the microstructures by promoting the formation of interdendritic MC carbides demonstrated as very useful for the mechanical resistance at elevated temperature (such as creep-resistance), solutions must be found to improve ductility and toughness. This supposes complicating the chemical composition with the introduction of new elements.

Despite the identified problems of room temperature behavior remaining to be solved, the demonstrated feasibility of such alloys by foundry, the characteristics of the as-cast microstructure and the high hardness allow being optimistic about the mechanical potential of the cast Cr-rich Nb-based alloys. This needs to be now verified by mechanical tests at high temperature. This is also true for their behavior in hot oxidation and corrosion. Thus, future work for understanding more and developing more this new family of high temperature alloys is substantial.

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