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Influence of Milling and Use of Ni and Al Containing Metal Binder in NbC-Based Cermets

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This study presents the development of niobium carbide cermets bound to nickel and Ni-12Al (wt%). The use of Ni-12Al (wt%) and Ni aims to replace strategic elements such as cobalt (Co) utilized in tungsten carbide-based cermets. Cermets of different compositions were processed by conventional powder metallurgy. Microstructural analysis with semi-quantitative chemical analysis by EDX, Vickers microhardness and density measurement were performed to evaluate the influence of high energy milling application and sintering temperature on the properties of these cermets. A milling time of 20 min in a planetary mill and sintering temperatures of 1420 ºC or 1450 ºC resulted in homogeneous microstructures, densities close to 90% and hardness of around 1000 HV_1, showing a potential for use of this material in cutting tools.

Keywords: *Cermets, Niobium Carbide, Nickel, Aluminum, Milling, Sintering.*

1. Introduction

Cermets are composite materials that can combine several properties of interest for applications in machining, such as high wear resistance, toughness[1] and good refractoriness. With certain compositions, cermets can reach hardness values close to hard metals and can also be applied in components or coatings which require, in addition to wear resistance, corrosion resistance[2,3].

One of the most outstanding cermets for application in cutting tools is WC-Co/Ni hard metals. However, there is an increasing interest in the replacement of WC, mainly due to the scarcity and high cost[4]. Additionally, WC/Co combination is considered a carcinogenic material to humans[5-7]. TiC or Ti(C,N) carbides with or without additions of other carbides were one of the explored in metal machining due to their high wear resistance[8-11]. Niobium carbides have been hardly used, although it was investigated in the sixties and seventies. Most probably because availability and prices at that time were prohibitive[12]. However, nowadays the situation is different, niobium has several indispensable applications, which made the metal available and at acceptable prices[12].

Niobium carbide (NbC) has high hardness (2004 HV) and higher melting point (3600 °C) than titanium (3420 °C) and tungsten carbide (2870 °C)[13]. The evaluation of the wear resistance of niobium carbides, without binders[14] and alloyed with 8% and 12% by volume of cobalt[15], showed superior wear resistance of these materials compared to different ceramics, cermets, carbide and hot spray coatings. NbC also had low solubility in steels and cast irons during metalworking[12,16], which can avoid adhesive and crater wear[8]. Furthermore, niobium is widely available in Brazil, highlighting the importance of this research[5].

Nickel and Ni-12Al (wt%) have high corrosion resistance at high temperatures[17-20]. Compared to Fe, the Ni binder resulted in a better combination of hardness and toughness in NbC cermets[21]. Cermets with 12 vol% of Ni or Ni-12Al (wt%) reached hardness close to 1300 HV_{10}. In NbC-based cermets, Ni-12Al (wt%) can inhibit NbC grain growth during liquid phase sintering[22]. Ni-12Al (wt%) powder is brittle and has low compressibility[23], however, this sintered intermetallic compound can reach a hardness of 600 $HV_{0.025}$ when forming the Ni_3Al phase[24]. Thus, it is assumed that the combination of Ni and Ni-12Al (wt%) as binder can contribute to more easily pressed cermets, in addition to improving their hardness.

Powder metallurgy is one of the most suitable techniques for the development of cermets, consisting of uniaxial pressing and sintering[8]. In the sintering step, a temperature suitable for the composition of the cermet must be chosen to obtain a consolidated part with the desired properties and characteristics. In liquid phase sintering, the binders promote the dissolution of carbides and reduce the porosity of cermets[22,25]. A high sintering temperature can increase the phase growth rate and lead to deformation due to excess liquid[26].

Research involving NbC, Ni and Al cermets is important, since the characteristics, properties and processing of cermets containing these elements still need further studies. The proper proportions of the constituents can give increased hardness, wear resistance and/or make processing easier. Therefore, the objective of this work was to analyze the hardness and microstructure of NbC-Ni-Al based cermets. These cermets were produced by conventional powder metallurgy, varying composition and processing parameters, such as milling and sintering temperature.

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2. Materials and Methods

2.1. Materials

The materials used to prepare the cermets were Ni, NbC and Al powders. The characteristics and manufacturers of these raw materials are shown in Table 1.

2.2. Preparing the Ni-12Al intermetallic

The intermetallic Ni-12Al (wt%) was prepared through high-energy milling of a mixture consisting of Ni elemental powders plus 12 wt% Al. Ni and Al powders with an addition of 1.5 wt% type C wax, as process control agent (PCA), were mixed in a Y-type mixer for 20 minutes. Subsequently, the mixture was subjected to milling in an argon atmosphere for 15 hours using an attritor mill, SAE/AISI 52100 steel balls, 400 rpm speed and ball/powder ratio of 20:1. After milling, the Ni-12Al powder was annealed at 1000 °C to ensure the formation of some amount of Ni_3Al. The annealing was carried out for 2 hours under an N_2H_2 atmosphere.

2.3. Cermet production

Conventional powder metallurgy (milling and pressing) was used in this research because it is a simple manufacturing method for obtaining NbC cermets and cutting tools. In addition, good mixing homogeneity and near-total density can be achieved by this method.

Cermets were manufactured with the compositions shown in Table 2, at 12 and 15 wt% binder, varying the contents of Ni and Ni-12Al. These powders were manually homogenized for 20 minutes, using a cylindrical holder containing 10 balls of SAE52100 steel with a diameter of 4.76 mm.

The cermets with 12 wt% binder were uniaxially pressed at 700 MPa and sintered under vacuum at 1350 ºC for 1 hour (heating rate of 5 ºC/min and cooling inside the furnace).

The NbC-7.5Ni-7.5(Ni-12Al) cermet was made with a higher binder content (15 wt%) to facilitate pressing. This mixture was milled in a planetary mill. This additional milling step was carried out to improve the distribution of cermet elements and minimize agglomerates which would impair the stage of pressing. Acrawax® C lubricant was added in the amount of 1.5 wt% to avoid delamination during pressing. The sintering temperature was also increased to provide greater diffusion of the components and consequently better properties[26]. Figure 1 summarizes the experimental methods to produce NbC-7.5Ni-7.5(Ni-12Al) cermet.

2.4. Material characterization

Ni-Al mixtures at different stages of preparation were characterized by X-ray diffraction (XRD). The Ni_3Al powder was also subjected to X-ray fluorescence (XRF) analysis to verify the presence of oxygen and to laser diffraction particle size analysis.

The phases of the sintered cermets were identified by XRD and SEM (Scanning Electron Microscopy) equipped with EDX (Energy Dispersive X-ray Spectroscopy) detectors. The XRD software was used to identify the phases formed.

Vickers microhardness tests ($HV_{0.25}$ - HV_1) were performed on consolidated samples of all studied compositions. The densities of the sintered samples were measured using Archimedes' principle, according to ABNT NBR 16661: 2017. The relative densities were calculated from the theoretical densities of the powders using the mixtures rule.

3. Results and Discussion

3.1. Ni-12Al (wt%) powder mixture

XRD spectra of Ni (ICSD 64089) and Al (ICSD 64700) powder mixtures at different stages of Ni-12Al production are illustrated in Figure 2. For the powders obtained by milling the mixture, the nickel peaks intensities were reduced, and their widths increased. The peaks shifted as there was (Ni) solid solution with Al and an intermetallic compound formation when the milling reached 15 hours[23,27-29].

Ni_3Al (ICSD 58039) and Ni (ICSD 64089) peaks were observed in the patterns of the milled and annealed mixture (Figure 2). As the Ni_3Al peaks had lower intensity, the (Ni) solid solution with Al[24,27,30,31] formed at the expense of the Ni_3Al

Table 1. Raw material characteristics.

Materials	Grain size (µm)	Purity (%)	Manufacturer
Ni	3.6 ± 0.4	99.8	Chemical JB Company
Al	39.7 ± 0.4	99.7	Alcoa do Brasil
NbC	2.2 ± 0.2	99.0	CBMM

Table 2. Chemical composition of investigated NbC based cermets by XRF.

Cermets	Composition (wt%)		
	NbC	Ni	Ni_3Al
NbC-1.4Ni-10.6(Ni-12Al)	88	1.4	10.6
NbC-3Ni-9(Ni-12Al)	88	3.0	9.0
NbC-6Ni-6(Ni-12Al)	88	6.0	6.0
NbC-12Ni	88	12	-
NbC-7.5Ni-7.5(Ni-12Al)	85	7.5	7.5

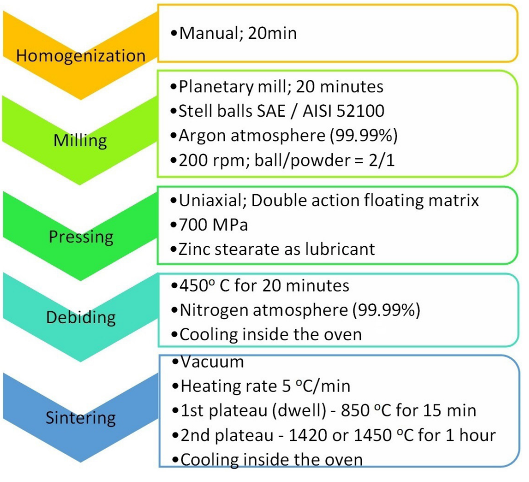

Figure 1. Powder Metallurgy processing parameters of the NbC-7.5Ni-7.5(Ni-12Al) cermet.

compound. However, a greater amount of the intermetallic Ni_3Al phase was expected due to the isothermal reactions with ignition that can occur during milling in attritor[29].

Figure 3 shows the morphology of the Ni-12Al powder mixture. The particles of this powder were clustered (Figure 3), which is typical in mechanical alloying. After annealing (Figure 3b), the particles continued to be agglomerated. The Ni-12Al powder produced was very fine and easily agglomerated. The average size of the annealed powder particles was 29.912 µm for 90% of the analyzed material volume (D_{90} in Table 3).

Oxygen was not found in milled and annealed Ni-12Al powder when submitted to X-ray fluorescence analysis, its composition was 90,0% Ni, 8.4% Al, 0.4% S, 0.3% P, 0.3% Sb_2O_3, 0.3% Cs, 0.1% Ca, 0.1% Y and 0.1% Fe by weight.

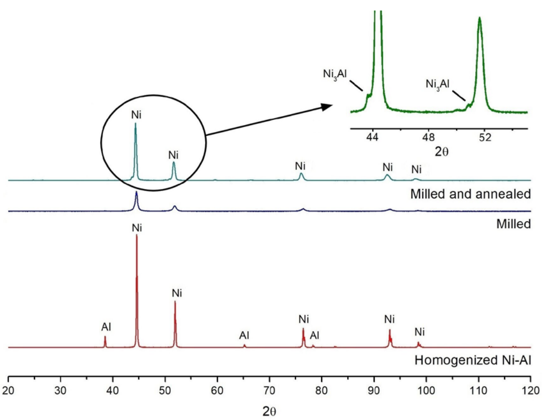

Figure 2. XRD patterns of Ni and Al powder mixtures after different processing stages to obtain Ni-12Al (wt%).

3.2. Cermets with different compositions

Figure 4 shows micrographs of cermets with different compositions and 12 wt% binder sintered at 1350 °C. The increasing content of the Ni-12Al compound increased the agglomerates, showing that this phase was not well distributed in the material. Agglomerates can be reduced by improving the material homogenization by milling[19].

The NbC-6Ni-6(Ni-12Al) cermet (Figure 4c) had a microstructure with fewer agglomerates. Therefore, the research continued investigating in greater detail on the binder containing Ni and Ni-12Al in the same proportion, NbC-7.5Ni-7.5(Ni-12Al) cermets. The increased binder (to 15 wt%) was to improve the densification. It is worth noting that in the NbC-7.5Ni-7.5(Ni-12Al) cermets, a lubricant was added to the powder mixture and an additional milling step was performed.

After sintering, NbC-7.5Ni-7.5(Ni-12Al) cermet had NbC (ICSD 618465) and Ni (ICSD 64989) phases as shown by the XRD pattern in Figure 5. According to the Al-Ni phase diagram[32], for 6.5 wt% Al (Al content in this binder), the microstructure at 400 °C consists of Ni_3Al and a (Ni) solid solution with approximately 4 wt% Al. The Ni_3Al compound was probably not detected by XRD due to its low concentration.

The microstructure of this cermet processed by powder metallurgy had a NbC matrix connected to a the (Ni) binder, as shown in Figure 6. The NbC grains were faceted with slight rounding of the edges and formed a highly interconnected carbide network with some NbC grains merged. The presence of Nb and Ni was confirmed by EDX semi-quantitative analysis, shown in Figure 7 and Table 4. Aluminum was not identified by EDX, probably due to its low content, although the aluminum content in the powdered cermet was 0.9 wt%. In cermets NbC-12vol%(Ni-12wt%Al), Nb was confined to the carbide phase and both Al and Ni, as well

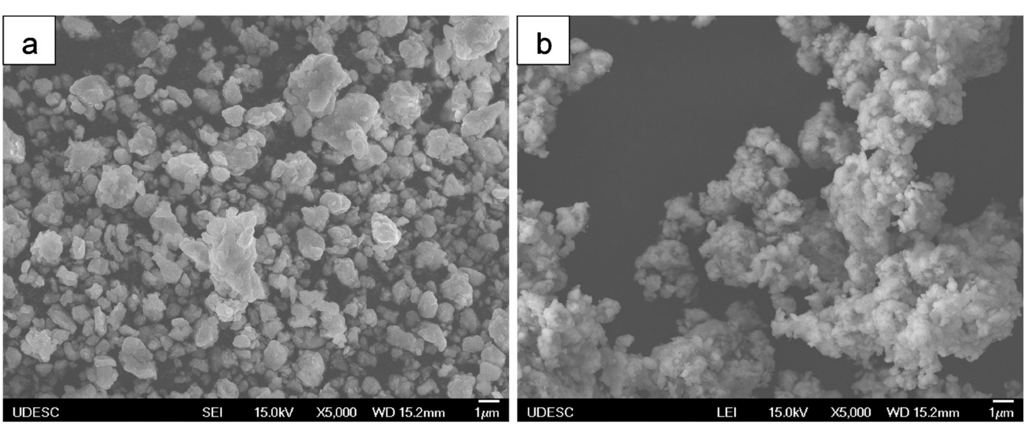

Figure 3. SEM – SE image of morphology of the Ni-12Al (wt%) powder mixture in (a) milled and (b) milled and annealed conditions.

Table 3. Cumulative particle size distribution of Ni and Al powder mixtures after different processing stages to obtain Ni-12Al (wt%).

Powder	D_{10} (µm)	D_{30} (µm)	D_{50} (µm)	D_{70} (µm)	D_{90} (µm)
Ni-12Al (milled)	1.140	1.984	3.195	5.623	12.201
Ni-12Al (milled and annealed)	1.421	2.635	4.408	7.999	29.912

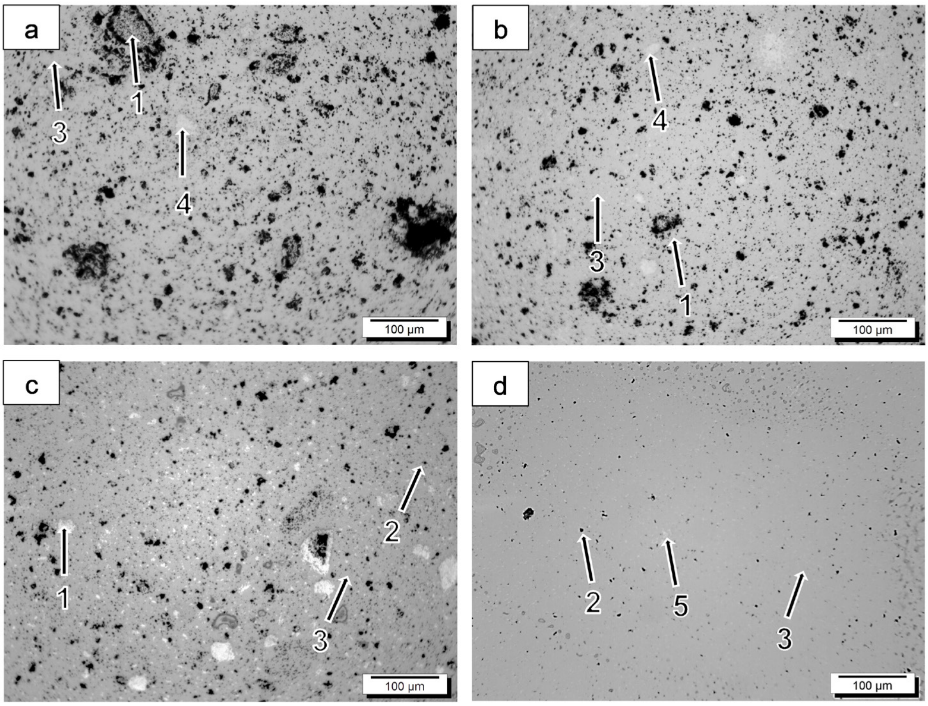

Figure 4. Optical microscopy images of NbC cermets with 12 wt% binder sintered at 1350 ºC: (a) NbC-1.44Ni-10.56(Ni-12Al), (b) NbC-3Ni-9(Ni-12Al), (c) NbC-6Ni-6(Ni-12Al) and (d) NbC-12Ni (wt%). 1 – Agglomerates, 2 – pores, 3 – NbC, 4 – (Ni), 5 – Ni.

as C were present in the binder phase according to WDS elemental mapping[21].

Figure 6 shows some regions may be Al_2O_3, while others are pores. Although no oxygen was identified in the Ni-12Al powder produced and the cermet NbC-7.5Ni-7.5(Ni-12Al) (Figure 7 and Table 4). These Al_2O_3 regions were also identified in NbC-12vol%(Ni-12wt%Al) cermets[22]. The presence of Al_2O_3 can be either due to Al_2O_3 impurities in the Al starting powder, or the Al reduction of residual Nb_2O_5 at the surface of the NbC starting powder[21], or the oxidation of a certain amount of Al occurred during lubricant removal from the compact, due to the presence of humidity in the purge gas[29].

By microstructural analysis (Figure 6) and since the melting temperature of Ni_3Al is 1383 ºC[33] it was deduced that Ni_3Al melted during sintering at 1420 °C or 1450 °C. Aluminum dissolved in Ni forming a the (Ni) solid solution. As Ni dissolves approximately 4 wt% Al according to the equilibrium diagram[32], a certain amount of free Al would have been left, which could have formed Al_2O_3.

The increased sintering temperature caused the growth of (Ni) and NbC phases (Figure 6). The (Ni) phase was approximately 53% larger at 1450 ºC (Figure 6e) compared to that of 1420 ºC (Figure 6b). Therefore, a higher sintering temperature led to greater diffusion and consequent increased phases size, which can reduce the material's hardness[22].

The consolidated NbC-7.5Ni-7.5(Ni-12Al) showed no dimensional distortions after sintering at 1420 ºC for 1 hour. On the other hand, sintering at 1450 ºC for 1 h caused dimensional deformations and delamination. Most likely, the rise in the sintering temperature led to the formation of an excess liquid phase, distorting the sample[26].

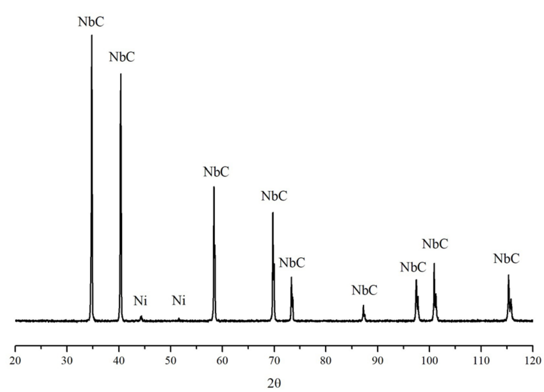

Figure 5. XRD pattern of the NbC-7.5Ni-7.5(Ni-12Al) cermet sintered at 1420 ºC.

Figure 8 shows the NbC-7.5Ni-7.5(Ni-12Al) cermet sample´s surface, where polygonal NbC particles with binder between then are shown. Semi-quantitative analysis through EDX also confirmed these phases (Table 5). The union of NbC particles is made by the binder during the sintering stage.

3.3. Hardness and density

The average hardness of the manufactured cermets reached approximately 1100 HV, as in Figure 9. The 12 wt% binder cermets had similar hardness for all compositions as shown in Figure 9 (yellow bars), with 95% confidence confirmed by the Student´s t-test. Although there was a greater variation of

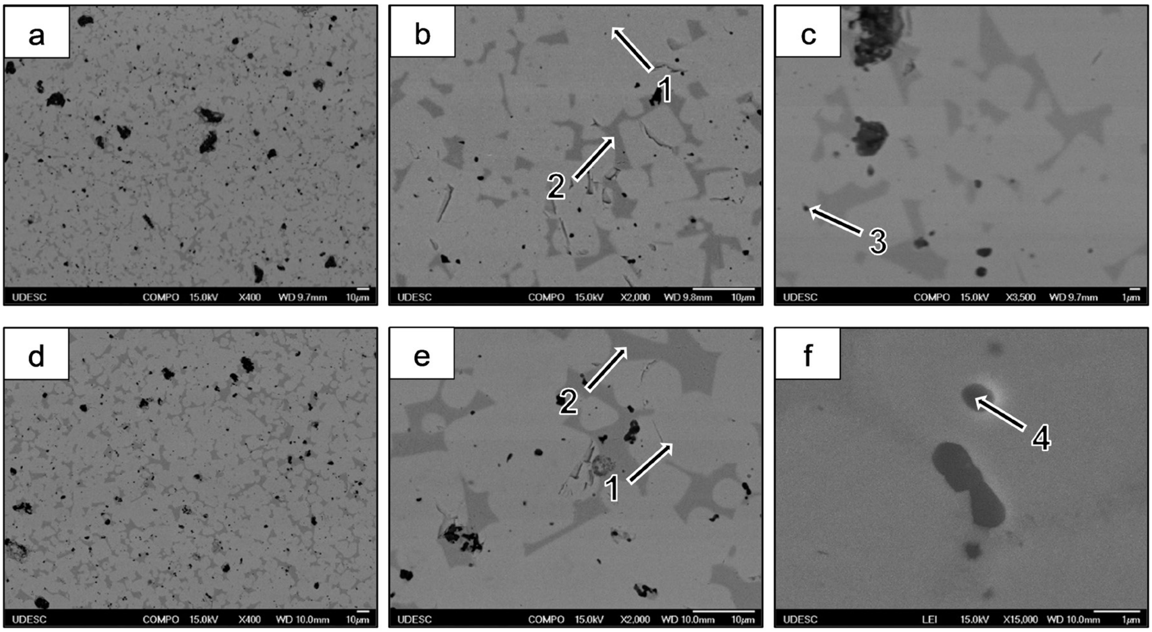

Figure 6. SEM – BSE images of the NbC-7.5Ni-7.5(Ni-12Al) cermet sintered at 1420 ºC (a-c) and 1450 ºC (d-f). 1 - NbC, 2 – (Ni), 3 - Al_2O_3, 4 - Pores.

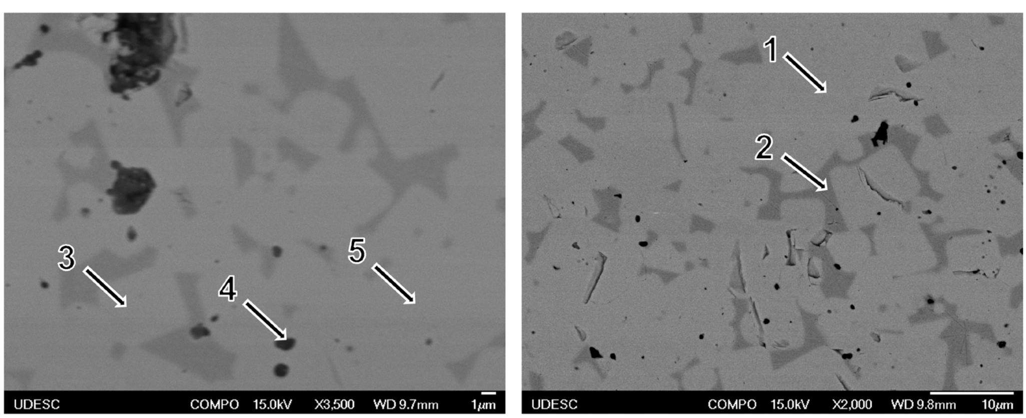

Figure 7. SEM – BSE image of NbC-7.5Ni-7.5(Ni-12Al) cermet sintered at 1420 ºC. 1, 3 and 5 – NbC, 2 – (Ni), 4 – Pore.

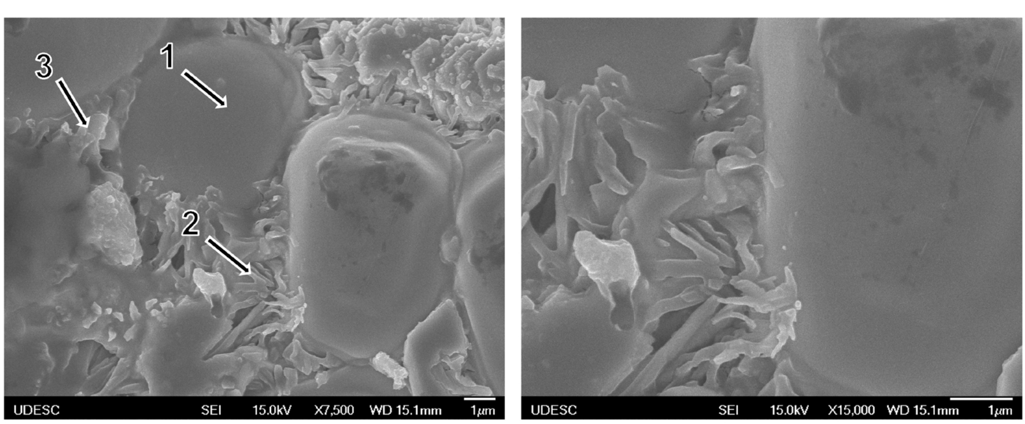

Figure 8. SEM – SE image of NbC-7.5Ni-7.5(Ni-12Al) cermet surface sintered at 1420 ºC. 1 - Polygonal NbC particles, 2 and 3 - Branches of the binder.

Table 4. EDX analysis of selected phases of the NbC7.5Ni7.5(Ni-12Al) cermet (Figure 7).

Phase	Chemical composition			
	Nb	Ni	Al	Another
	wt%	wt%	wt%	wt%
1	100.0	-	-	-
2	-	45.7	-	5.7
3	100.0	-	-	-
4	55.1	44.2	0.7	-
5	80.3	19. 7	-	-

Table 5. EDX analysis of selected phases of the NbC7.5Ni7.5(Ni-12Al) cermet surface (Figure 8).

Point	Phase	Chemical composition			
		Nb	Ni	Al	Another
		wt%	wt%	wt%	wt%
1	NbC	86.9	2.0	0.2	10.9
2	(Ni)	17.8	29.2	7.0	46.1
3	(Ni)	39.8	26.4	4.9	28.9

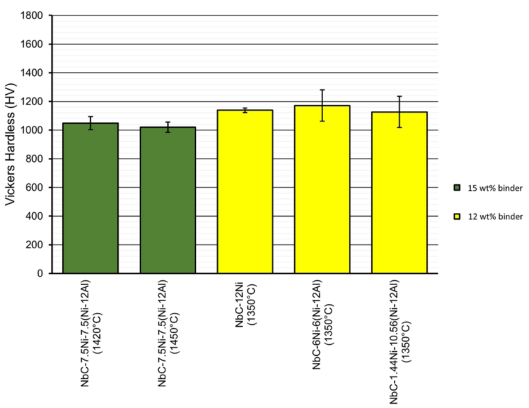

Figure 9. Hardness of NbC cermets.

hardness values in samples containing (Ni) phase that could have resulted from the agglomerates (Figure 4).

In the NbC-7.5Ni-7.5(Ni-12Al) cermets, there was less variation in hardness (Figure 9) than in the NbC-6Ni-6(Ni-12Al), which can be attributed to the milling[19] or/and the higher sintering temperature[26], both could lead to greater microstructural homogeneity and consequently less variation in hardness. Furthermore, the sintering temperature did not affect the hardness of the NbC-7.5Ni-7.5(Ni-12Al) cermets, since these values were statistically the same for the cermets sintered at 1420 ºC and 1450 ºC (Figure 9).

In addition, NbC-7.5Ni-7.5(Ni-12Al) cermet sintered at different temperatures exhibited close density values, with a difference of 2.8%. The cermet sintered at 1450 ºC reached a density of 7.2 ± 0.2 g/cm^3 (relative density of 92%), while the cermet sintered at 1420 ºC reached a density of 7.0 ± 0.1 g/cm^3 (90%).

Cermets with binder proportions similar to this work, with binders of Ni, Co, Ni-Al or Fe-Al had good densification and reached hardness between 1200 HV_{10} and 1400 HV_{10}, produced by isostatic compaction[22]. The cermets of the current work made by uniaxial compaction and sintering reached hardness close to isostatically pressed samples[22]. Thus, the investigated cermets were promising, as they could achieve better properties (density and hardness) if they were processed by a more advanced technique, like Field Assisted Hot Pressing that achieves full density[2].

4. Conclusions

The microstructure of the NbC-7.5Ni-7.5(Ni-12Al) cermet consisted of regions of NbC connected by a (Ni) solid solution.

The NbC-7.5Ni-7.5(Ni-12Al) cermets had average hardness between 1020 and 1049 HV and relative densities of approximately 90%, values close to previous work.

The presence of Ni-12Al (wt%) in the binder increased the hardness in some regions of the cermet, shown by the range of hardness values of the 12% binder cermets.

Even being processed by a simple technique, uniaxial compaction and sintering, the cermets of this work exhibited good properties, showing them to be promising materials.

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