The Effects of Sinter-Hip Processing on the Machining Performance of Cemented Carbides Based on NBC-Ni Systems as Alternative Cutting Tools

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Abstract

This work aims to investigate the technical feasibility of using sintered carbides based on NbC-Ni substitute of the WC-Co class, adding the Sinter-Hip step in the sintering process, with 20 bar pressure nitrogen gas. The samples were produced investigating two process variables: sintering cycles and chemical composition. The samples were qualified in terms of Vickers hardness, toughness and density by Archimedes. The machining experiments were carried out on quenched and tempered AISI 4340 steel material. Flank wear progression was defined as the control parameter, analyzing wear via CCD camera, stereoscopic magnifying glass and SEM/EDS. The best result obtained the binding phase content 10%w, when compared to a commercial WC-Co insert. It showed superior performance at 88% of the average life of the cutting edge. However, the dispersion of this lifetime was higher than the WC-Co reference tool, 15%. The samples of NbC-10%p Ni showed a higher level of porosity than WC-Co. For better densification, new samples with the same composition were produced by adding the Sinter-HIP step. New tests were carried out and the results showed that the adjustment of the sintering process was fundamental. Densification and microstructure alteration reduced the dispersion of the average lifetime of this new NbC-Ni class.

1. Introduction

Niobium carbide has been used as a secondary hard phase material in high speed steels, tungsten carbide and titanium cermet tools, but its potential as a primary hard phase in cutting tools needs to be further explored. The hardness of niobium carbide is comparable to that of titanium, tungsten, molybdenum and chromium carbides [1], as shown in Fig. 1. Its high hardness offers high wear resistance, which is desirable for cutting tool applications. On the other hand, its low solubility and high sintering temperatures are the two main obstacles to be overcome. The improvement of the sintering technique makes it possible to exploit its full potential as a material for cutting tools. The process of hot compaction, high frequency induction sintering and plasma sintering (SPS) enables high quality NbC-based carbides to be obtained [1, 2].

Cutting tools are subject to enormous stresses, high temperature, high contact pressure and different wear and damage conditions during chip formation. The main desirable requirements for cutting tool materials are hardness and pressure resistance, bending strength and toughness, edge strength, internal bond strength, high temperature resistance, oxidation resistance, low diffusion and adhesion propensity, strength to abrasion and reproducible wear behavior [4].

According to Peter and Rosso [5] an effective cutting tool must have good wear resistance to be able to maintain its integrity against abrasion during service, an adequate toughness to be able to absorb the energy imposed by different loading / unloading forces and vibrations, and adequate hardness to be able to survive continuous changes in shape and geometry during cutting.

An ideal cutting material combines high hardness with toughness and chemical stability. The problem is that hardness and toughness represent opposite properties; normally, as the hardness value increases, the toughness value decreases. As a result, there is no single cutting material which achieves all three conditions simultaneously. Therefore, the biggest challenge in the development of cutting tools is finding the balance between these three conditions [4, 6, 7, 8]. The development of cutting tool materials should consider these requirements as a balanced compromise, since there is no cutting tool material that combines all the properties at their optimum level.

This work aimed to analyze the effects of the addition of the Sinter-Hip step in the manufacturing process of NbC-Ni based cutting tools, compared to the same grade which had previously been produced in the vacuum oven plus argon, with 20 bar pressure for rapid cooling from 1420°C to 800°C. These tools were compared to a WC-Co P10 commercial insert in turning tests on quenched and tempered AISI 4340 steel, hardness 320 ± 20 HB. Flank wear progression was used as a benchmark to determine wear resistance performance.

2. Cemented Carbides Inserts For Cutting Tools Machining Operations

2.1. NbC-Ni based cemented carbide tools

Niobium carbide (NbC) is used as the secondary phase carbide to enhance wear resistance while improving hot hardness and limiting grain growth. However, recent research has shown that niobium carbide has tribological, physical and mechanical properties important for cutting tool applications that can be used in place of tungsten carbide, which has long been used in this application [1, 2].

Recently, special attention has been given to the use of niobium for cutting tool applications, using niobium carbide (NbC) as the main hard phase of the cutting tool substrate. Some of the properties of niobium carbide have been compared with those of tungsten carbide. Niobium carbide has some distinct advantages over this established cutting material. However, the mechanical and particularly tribological properties of NbC remain largely unexplored [1, 2].

The reduction in the world supply of W and the consequent increase in the prices of Co, which are the main elements for manufacturing the substrate for cutting tools, continue to incentivize scientific research for the development of new materials for cutting tools [9, 10].

Several studies are being carried out into the development of products with the addition of niobium: steels for on-shore piping for oil as well as gas pipelines that work in more extreme environments; niobium microalloyed steels for weight reduction of products; orthopedic prostheses with niobium-titanium and titanium-niobium-zirconium alloys by additive manufacturing; cermets and niobium-based coatings for applications in tools, nickel-based superalloys and alloys for applications at high

temperatures [1, 11, 12, 13, 14, 15]. All these recent studies have made it possible to propose a new approach in the sintering process for the fabrication of NbC-Ni based cutting tools.

Niobium, in recent years, has received greater attention, with regard to sustainability (environment). Niobium oxide (Nb_2O_5) and niobium carbide (NbC) do not present risks, or critical notifications, to the Registration, Evaluation, Authorization and Restriction of Chemical Substances Program (REACH), which is regulated by the European Union with the aim of improving the protection of human health and the environment from the risks posed by chemicals. Niobium carbide has not been constrained by any critical assessment to date. On the other hand, compositions used to obtain WC-Co powder through the reduction process, such as tungsten trioxide (WO_3) and tricobalt tetroxide (Co_3O_4), have received several classifications due to mutagenic problems, carcinogenic toxicity by REACH [1, 2, 11, 13].

2.2. Vacuum sintering under protective gas

The sintering process takes place under vacuum or under protective gas at the appropriate temperature for the material. A defined and reproducible sintering atmosphere without atmospheric oxygen is important. This is due to several factors, such as impurities, lack of lubricant homogenization, insufficient compaction pressure, gas removal, insufficient densification. At certain pressures and temperatures, the process gas supply during sintering with argon, hydrogen, nitrogen, methane, carbon dioxide, and others can influence the structure and chemical composition of workpieces. The sintering vacuum or under protective gas (up to 0.1 MPa) is suitable for a high level of densification with little or no apparent porosity [16].

2.3. Sinter-HIP process

According to German [17], sintering has difficulty in densifying materials that naturally resist high temperatures. Typically, the driving force for sintering comes from the surface energy associated with small particles. External pressure increases the driving force, leading to faster densification. Thus, pressure-assisted sintering is a means of densifying materials that are normally resistant to sintering.

Currently, where the high performance of advanced materials is required in engineering applications, metallic and ceramic powders must be non-porous (full density). The main hot consolidation technologies currently applied in the industry for powder consolidation are Hot Pressing (HP), Hot Isostatic Pressing (HIP), Powder Extrusion and Powder Forging [18]. In cemented carbide industries, another technique has been used commercially, called Sinter-HIP.

Sinter-HIP is a thermal consolidation method for cemented carbide in which the simultaneous application of heat and pressure (3 to 10 MPa) fully consolidates the carbide during the sintering process. Following the vacuum sintering process, argon or nitrogen gas is introduced into the furnace at the sintering temperature to further reduce porosity and ensure material quality for high-performance carbide tools and other sintered parts. It results in a product which contains little or no porosity, producing a component as close as possible to the theoretical total density [17].

Vacuum sintering and filling the sintering furnace with pressurized gas to aid final densification are employed in Sinter-HIP processing (a pressure-assisted sintering process). A typical cycle is shown schematically in Fig. 2.

There have been several studies comparing the vacuum sintering process with the Sinter-HIP and the HIP process for manufacturing inserts or cutting tools for Cermets based on tungsten carbide. In these works, they show an increase of up to 100% in the resistance properties and there are no “lakes” of the binding phase in the homogeneous microstructure when compared to the HIP process. These studies show that for material cutting tools, in cases with a low percentage of binder, the increase in the strength of Sinter-HIP alloys is higher than when the materials vacuum is sintered using the Sinter-HIP process [19].

3. Materials And Methods

3.1. Development of NbC-Ni cemented carbide tools by vacuum sintering under argon

Cutting tools of different grades of NbC-Ni were developed by varying the percentages of mass of NbC and Ni and the sintering variables (furnace atmosphere, temperature, and threshold time). The best result obtained in the machining process was with the NbC-Ni class with a binder content (Ni) of 10% by weight - NbC-Ni_967 [20]. Table 1 presents the characteristic of the NbC-Ni_967 class: composition, hardness, fracture toughness, sintering furnace atmosphere, threshold time, sintering temperature, density, and relative density. The commercial grade WC-Co – P10 (K_{IC} : $10.3 \pm 0.1 \text{ MPa}\cdot\text{m}^{1/2}$ and hardness: $1670 \pm 0.5 \text{ HV}_{30}$) was used as a reference in the machining tests.

Initially, the development of the NbC-Ni-based grade used the vacuum sintering cycle, but to reduce nickel losses during sintering, a dynamic argon atmosphere was introduced for the sintering cycle, as can be seen in Fig. 3. The use of argon minimized nickel evaporation and promoted a better control of the nickel content after sintering [20]. The sintering temperature and threshold time were varied for the development of insert grades based on NbC-Ni. This allowed variations in the mechanical properties of the developed cutting material classes to be obtained. With the variation of threshold time, there was a variation in the hardness of the material, decreasing mainly due to grain growth, or increasing due to the dissolution of niobium in nickel. The behavior of fracture toughness was the inverse of hardness, as with increasing hardness there is a decrease in fracture toughness. A compromise solution was reached between these two phenomena. The evolution of flank wear was selected as a control parameter to evaluate the performance of the NbC-Ni cutting tools developed in comparison with the reference WC-Co [20].

Table 1

Composition, hardness, fracture toughness, sintering furnace atmosphere, threshold time, sintering temperature, density, and relative density for the NbC-Ni_967 tool

Hardness (HV ₃₀)	K _{IC} (MPa.m ^{1/2})	Sinter atmosphere	T _{sinter} (°C)	t _{sinter} (min)	d (g/cm ³)	d _r (%)
1,460 ± 40	8.9 ± 0.4	Vacuum + argon	V – 1,380 Ar – 1,420	20	7.60	97.9
Composition (%wt): 10%Ni + 12%WC + 14%TiC + 64%NbC						

First of all, the dilatometry test was performed, using the 402S Netzch equipment. A sample was produced and placed inside an alumina tube, Fig. 4 (a), and this was placed inside a furnace with a dynamic nitrogen atmosphere with a flow rate of 200 ml/min. With the definition of each sintering cycle, the linear contraction measurement was carried out. Figure 4 (b) schematically shows the achievement of linear contraction using a dilatometer.

The NbC-Ni alloy started to contract around 1200°C, so the sintering could still be considered to occur in the solid state. At a temperature of 1300 °C, the alloy reached almost 15% of its linear contraction, for a 60-minute stabilization. With the rapid rise from 1300°C to 1380°C, the liquid sintering phase reached its contraction of approximately 20%.

During heating, as seen in Fig. 4(b), the samples underwent contractions and shrinkage rates, significant changes in dimensions, due to phase melting near 1280°C. Furthermore, the dissolution of NbC crystals and probably WC crystals led to pore closure and a better distribution of the Ni binding phase on the surfaces of WC and NbC particulates. This type of dilatometer uses two samples of the same material. One is the reference sample, which is dense so that the detection probe can produce physical alteration of the specimen, Fig. 4(b).

These changes provide information about the sintering mechanisms of these composites (liquid and solid phase sintering), which must be accompanied by the observation of microstructures under an optical microscope. It can be said that, in general, the higher the sintering temperature, the greater the densification, and the greater the linear and volumetric contraction of these materials. It can be said that in the shrinkage process, the sintering of the solid and liquid phase plays an important role in the densification of the samples.

3.2 Development of NbC-Ni-based carbide tools by vacuum sintering under argon

After all the analyses performed on the previously produced NbC-Ni classes, two new NbC-Ni samples were developed adding the Sinter-HIP step: the SH1 and SH2 classes had as reference the NbC-Ni_967 class that obtained the best performance in relation to NbC-Ni classes. The addition of the Sinter-HIP

turning process, maintaining or improving the performance achieved in the machining tests performed in the grade NbC-Ni_967.

The sintering process consisted of two steps. The first was the same as the process performed for the NbC-Ni_967 grade and described in Table 1, vacuum sintering with argon. And the second step was the Sinter-HIP process, the parameters of which are described in Table 2.

The only change in powder preparation between the NbC-Ni_967 class in relation to the SH1 and SH2 class was the change in milling time from 0.5 hour to one hour in order to obtain a more homogeneous structure (attrition mill). All the other preparation steps remained the same. Grades SH1 and SH2 were produced at the same time and machining tests took place simultaneously for both grades.

The first sintering step, under vacuum + argon, for these new grades was the same as the NbC-Ni_967, Fig. 2. The second step, the Sinter-HIP process, was added after the first step. The cycle used in resintering Sinter-HIP is shown as Fig. 5. The main difference between cycles SH1 and SH2, shown in Fig. 5(a) and Fig. 5(b) respectively, were that for class SH1 after N₂ injection and pressure at 20 bar the furnace was turned off, while in cycle SH2 the furnace was kept on for the temperature drop.

Table 2
Composition, hardness, fracture toughness, sintering furnace atmosphere, sintering temperature, density, and relative density for the SH tools – Sinter-HIP

Samples	Hardness (HV ₃₀)	K _{IC} (MPa.m ^{1/2})	tsinter (°C)	d (g/cm ³)	d _r (%)
SinterHip1 (SH1)	1,402 ± 25	9.2 ± 0.3	1,400	7.69	99
SinsteHip2 (SH2)	1,407 ± 29	9.0 ± 0.3	1,400	7.73	99.5
Composition (%wt)	10%Ni + 12%WC + 14%TiC + 64%NbC				
Atmosphere	Vacuum + N ₂ + pressure: 20 bar				

The development of these two new grades was based on the analysis of the machining performance, in particular the dispersion of the cutting-edge lifetime and the porosity of the NbC-Ni samples in relation to the WC-Co reference sample. Through these analyses it was decided to manufacture the NbC-Ni_967 classes with the addition of the Sinter-HIP process (SH1 and SH2), as this class was the one which had obtained the best machining performance result when compared to the WC-Co reference sample.

The evolution of flank wear was performed using turning experiments. The end of life of the cutting edge was considered for a Flank Wear V_B: 0.3 mm. The experiments were carried out under the same conditions as the previous tests: cutting speed V_c: 160 m/min; feed rate f: 0.1 mm/rev; cutting depth a_p: 1.5 mm and cutting fluid was used [20]. The specimens were the same as the ones used in the previous experiments: AISI 4240 steel quenched and tempered, Ø90 x 325 mm; hardness: 320 ± 20 HB [20].

Comparisons were also made in relation to microstructures (carbide distribution, grain size and porosity) and the plasticity of the binder, checking to see if there were changes with the addition of the Sinter-HIP process.

Porosity analysis of the different samples was performed based on Standard B276-5 (2015) Test Method for Apparent Porosity in Cemented Carbides [21]. There are three types of porosity classification in this standard:

- Type A: pores up to 10 μm analyzed at 200x magnification.
- Type B: pores in the range from 10 to 25 μm analyzed at 100x magnification.
- Type C: classify porosity resulting from uncombined carbon analyzed with a magnification of 100 or 200x.

The characterization of the plasticity of the binder of the classes of cutting tools based on NbC-Ni and WC-Co reference were carried out using indentation tests instrumented by the Hyston triboindenter (TI-950). In these tests, the load-displacement curves were obtained, as shown in Fig. 6. The results were analyzed by specific algorithms, which provide instrumented hardness and reduced modulus of elasticity.

The experimental conditions for the characterization of Young`s modulus of the materials analyzed are described in Table 3. The preparation of the instrumented indentation experiments on the samples was a function of the applied load and the test time. All indentations were performed with a Berkovich diamond indenter.

Table 3
Experimental conditions of nanoindentation tests to obtain the Youngs mod *us* and *b* ∈ *ders* hardness of samples

Loads (μN)	Loading Time (s)	Creep Time (s)	Unloading Time (s)	Number of Replications
300	5	2	5	10

Based on the reduced modulus of elasticity of the binder, it was possible to calculate the modulus of elasticity of the samples using the Oliver and Pharr equation [22].

$$\frac{1}{E_r} = \frac{(1 - \nu_i^2)}{E_i} + \frac{(1 - \nu_s^2)}{E_s} \tag{1}$$

In this, E_r is the reduced modulus; E_i, and ν_i and E_s, ν_s are Youngs mod *us* and *Poissons* ratio of the diamond indenter and the specimen, respectively.

Statistical analysis of all data obtained during the experiments was performed. A 95% confidence interval Loading [MathJax]/jax/output/CommonHTML/jax.js

this interval can overlap in several cases, it was necessary to perform a statistical analysis to compare the means of the analyzed variables. To confirm whether there was a statistical difference between the means, an ANOVA analysis of variance was used, through the Minitab software, in order to confirm the differences between the analyzed variables, between the classes of the NbC-Ni tools and between them and the class reference of WC-Co.

4. Analysis Of Results

4.1. Porosity and microstructure analysis

Figure 7 presents the microstructures of the analyzed samples and Table 4 presents the result obtained in this analysis.

Table 4
Classification of porosity types - SH classes compared to NbC-Ni-967 and WC-Co reference classes

Types	SH1	SH2	NbC-Ni_967	WC-Co reference
Type A	A4	A4	A6	A2
Type B	—	—	B2	—
Type C	—	—	—	—

There was a reduction in porosity of SH classes compared to NbC-Ni-967. With the addition of Sinter-HIP there was an elimination of Type B porosity (> 10 to $\leq 25 \mu\text{m}$) and an improvement in Type A porosity (pores $\leq 10 \mu\text{m}$), but the results are lower than the WC-Co reference class. In the analyses carried out previously, all NbC-Ni classes, produced in vacuum sintering and vacuum sintering + argon, had presented Type B2 and B6 porosity and Type A6 porosity [21].

Classes SH1 and SH2 presented “Ni lakes”, and the same had occurred for the previously developed classes, Fig. 6. The distribution of carbides (classes SH1 and SH2) remained heterogeneous, even doubling the milling time, confirmed by the distribution of tungsten in Fig. 8 (a) (b) (c), which show tungsten concentration in regions of area “B”, compared to regions of area “A”.

Comparing the microstructures of SH1 and SH2, it is possible to observe, in Fig. 9 (d) (e), that the grain size of the SH2 class carbides is smaller than that of the SH1 class. This could be due to the cooling time in the sintering process. Class SH2 had a longer cooling time, as the Sinter-Hip oven was kept on during this process, while for Class SH1 the oven was turned off, resulting in a greater drop in temperature during this process.

Two phenomena occurred for the differentiation of grain size between classes SH1 and SH2: the dissolution of secondary carbides, which restricts the growth of NbC grains, and the longer time, which Loading [MathJax]/jax/output/CommonHTML/jax.js by coalescence. From the result obtained, as shown in Fig. 9

(d) (e), the first phenomenon prevailed over the second, as the grains of SH2 are smaller than those of SH1, even with the cooling time in the sintering process of SH1 being shorter than that of SH2.

Therefore, the microstructure of SH classes, even with the reduction of porosity, remains heterogeneous compared to the WC-Co reference class.

4.2. Binder plasticity analysis

In previous tests [23], the class which had the lowest modulus of elasticity of the binder was WC-Co reference to 192.2 GPa, while the class NbC_Ni-967 showed the lowest elastic modulus of the Ni-NbC classes (235.7 GPa). There was a variation between 235.7 and 367 GPa of the analyzed NbC-Ni classes [23]. In relation to the hardness of the binder (H) the NbC-Ni classes presented a variation between 3.93 and 5.87 GPa, while in the NbC-Ni_967 class the hardness was 4.70 GPa, but statistically equal to the WC-Co reference, which was 4.25 GPa [23].

It was possible to observe that classes SH1 and SH2 had the same behavior as the analyzed parameters. The two classes produced with the addition of the Sinter-HIP process had the same statistical behavior in relation to the NbC-Ni_967 class, but the dispersion of the results of the analyzed parameters was much smaller in the SH classes when compared to the NbC-Ni_967 class, as can be seen in Table 5. This may indicate that the addition of Sinter-HIP reduced the variability of the results of the analyzed parameters.

Table 5
Average parameters obtained via nanoindentation: SH classes compared to NbC-Ni_967 and WC-Co reference classes

Variables	SH1	SH2	NbC-Ni_967	WC-Co reference
H (GPa)	5.03 ± 0.18	4.97 ± 0.21	4.70 ± 0.71	4.25 ± 0.21
E (GPa)	233.3 ± 19.4	232.0 ± 24.6	235.7 ± 35.9	192.2 ± 16.0

With the introduction of the Sinter-HIP process, there was a reduction in the dispersion of the results of classes SH1 and SH2 in relation to class NbC_Ni-967. There was therefore a greater differentiation of results between classes SH in relation to WC-Co reference than the classes NbC_Ni-967 and WC-Co reference.

4.3. Machining analysis

Machining experiments were carried out with classes SH1 and SH2 under the same conditions as the previous tests. Figure 10 shows the comparative performance of each class developed in the Sinter-HIP process (SH1 and SH2) compared to the WC-Co reference and NbC-Ni_967, which was the class that had presented the best result in the machining tests previously, in terms of average lifetime of the cutting edge for a defined flank wear ($V_B = 0.3$ mm).

The grades sintered under vacuum and argon with the addition of the Sinter-HIP process, SH1 and SH2, as of the lifetime of the cutting edge during the machining

process. Class SH1 had an average lifetime per cutting edge of 28.8 ± 2.1 minutes, while class SH2 had an average lifetime of 43.8 ± 1.6 minutes. Therefore, grade SH2 had a cutting edge lifetime greater than SH1 by 1.8x.

The main difference between these classes in the manufacturing process was the cooling time in the Sinter-HIP process. Class SH1 had a shorter cooling time with a greater drop in temperature compared to class SH2. Due to this difference in the process, the microstructures they showed were different, and the SH1 had larger NbC carbide grains than the SH2 class, Fig. 9 **(d)** **(e)**. Hardness and fracture toughness were also different, as described in Table 2.

Comparing the SH classes with the NbC-Ni_967 class, the SH1 class presented a 41% lower performance and the average lifetimes per cutting edge were, respectively, 28.8 and 48.9 minutes. The main difference between them was the hardness and fracture toughness; the SH1 grade had a hardness of $1,402 \pm 25$ HV₃₀ and $K_{IC} = 9.2 \pm 0.3$ MPa.m^{1/2}, while the NbC-Ni_967 grade had a hardness of $1,460 \pm 29$ HV₃₀ and $K_{IC} = 8.9 \pm 0.4$ MPa.m^{1/2}. However, the lifetime dispersion of the cutting edge of SH1 was 8%, while the NbC-Ni_967 was 15%, representing a dispersion reduction in the order of 3.5 times. The main difference between the two classes was the reduction in porosity of SH1 as shown in Table 4.

Class SH2 statistically showed equal performance to NbC-Ni_967, with 43.8 and 50.2 minutes respectively. However, there was a reduction in dispersion of 4.6 times, from 15–3.7% for the SH2 class. The hardness of class SH2 was lower than class NbC-Ni_967, $1,460 \pm 29$ HV₃₀ and $1,407 \pm 29$ HV₃₀ respectively. The fracture toughness was statistically equal; class SH1 was 9.0 ± 0.3 MPa.m^{1/2} and class NbC-Ni_967 was 8.9 ± 0.4 MPa.m^{1/2}. However, SH2 had a lower porosity level than NbC-Ni_967 (Type A: A4; Type B: no pores and Type A: A6; Type B: B2, respectively).

Comparing WC-Co grade reference with SH1 class showed a statistically equal performance in the average lifetime of the cutting edge of 25.9 minutes for the WC-Co and 28.8 minutes for the SH1 class. However, the dispersion of WC-Co was 2.6 times lower than SH1 (3.2% and 8.0% respectively).

Class SH2 outperformed the average lifetime of the cutting edge by 68.9%, 43.8 versus 25.9 minutes respectively. However, the dispersion of reference WC-Co was 3.2% while the SH2 was 3.7%. Therefore, with the addition of the Sinter-HIP step there was a reproducibility in the wear behavior. SH2 had a dispersion 1.15 times higher than WC-Co reference and 75.5% lower than NbC-Ni_967 while the NbC-Ni_967 class had a dispersion 4 times higher than the WC-Co reference as can be seen in Fig. 11 **(a)****(b)****(c)**.

Comparisons were made between the relative SH-Ni_967 NbC and WC-Co benchmark for the wear mechanisms that acted during the turning process, shown in Fig. 12.

Class SH1 showed more regular abrasive wear on the cutting edge on the main face compared to NbC-Ni_967. This could be due to the porosity reduction with the addition of the Sinter-HIP process in the SH1 class. Grade SH1 had less spalling on the edge of the edge. These spalls were due to adhesive wear that
Loading [MathJax]/jax/output/CommonHTML/jax.js /e wear caused more damage in the NbC-Ni class than in the

WC-Co class because for this system the physicochemical interactions between carbides and binder are different and the microstructure analysis showed that there is greater contiguity in the NbC-Ni classes than the WC-Co reference class.

Comparisons between the SH1 and SH2 grades with the WC-Co reference grade, with the addition of the Sinter-HIP process, show there was a reduction in adhesion wear mechanisms and a better uniformity of abrasion wear. However, compared with the WC-Co reference class, the abrasion is not as uniform as can be seen in Fig. 10. This may be due to the fact that the WC-Co reference class presents a more homogeneous microstructure than the SH classes. The SH classes have a lot of contiguity and a heterogeneous distribution of carbides.

However, regarding the adhesion mechanism, which is the main cause of spalling of the cutting edge, the SH2 class presented damage very similar to the WC-Co reference. While in the SH1 class the damage presented was greater than the reference class, there was less damage than the NbC-Ni_967 class, demonstrating that the addition of the Sinter-HIP process reduced the effect of the adhesion mechanism and left the abrasion wear more uniform.

5. Conclusions

The experiments carried out in this work aimed to evaluate the development of material grades based on NbC-Ni, produced by powder metallurgy, for application in cutting tools for machining, as an alternative to the traditional WC-Co. The performance of these tools, taking into consideration wear mechanisms and edge lifetime, was evaluated based on the process-structure-properties triad.

Considering its experimental context, the work allows us to present the following conclusions:

- With the addition of the Sinter-HIP process in the grades that were produced under vacuum and argon, there was a reduction in the dispersion in the life of the cutting edge compared to the NbC-Ni grades, which indicates that the Sinter-HIP process improved the microstructure and reduced porosity, presenting a less heterogeneous microstructure.
- The density of the grade that obtained the best machining performance, NbC-Ni_967, was 7.60 g/cm³. With the addition of the Sinter-Hip step for the manufacture of SH1 and SH2 classes, based on the NbC-Ni-967 class, there was greater densification, with the density for SH1 rising to 7.69 g/cm³ and to 7.73 g/cm³ for SH2.
- The NbC-Ni classes with the addition of the Sinter-HIP process did not present Type B porosity, but presented Type A4 porosity instead. Without the use of Sinter-HIP, the porosity ranged from Type B2 to B6 and from Type A4 to A6.
- The adhesion wear mechanism was significantly reduced with the addition of the Sinter-HIP step in the sintering process.
- The SH2 grade presented an adhesion level very similar to the WC-Co reference, which may mean

of the carbides with the binder had improved with the addition

of the Sinter-Hip process.

- Classes SH1 and SH2 have the same composition as class NbC-Ni_967. However, the results obtained in the nanoindentations were statistically equal (hardness and Young`s Modulus), but with a significant reduction in the dispersion of the analyzed parameters due to the insertion of the Sinter-HIP process.
- The hardness of the binder in the NbC-Ni and WC-Co grades was statically equal.
- The NbC-Ni grades with the lowest Young`s Modulus, 232 GPa, had the best machining performance in terms of the average life of the cutting edge (NbC_Ni-967 and SH2);
- The Young`s modulus and the hardness of the binder of the SH classes were statistically equal to those of the NbC-Ni_967 class. However, class SH1 had a cutting edge average life 44% shorter than NbC-Ni_967, while class SH2 was statistically equal to NbC-Ni_967. What differentiates SH1 and SH2 is the grain size between the two classes, with the microstructure of SH1 being coarser than that of SH2.
- All NbC-Ni classes presented “Nickel lakes”, regardless of the sintering process.
- The best combination of fracture toughness and hardness, considering the average life of the cutting edge, was:
 - K_{IC} : 8.9 to 9.2 MPa.m^{1/2}
 - Hardness: 1405 to 1460 HV₃₀
- Grades with the addition of the Sinter-HIP process have a lower dispersion of the cutting edge lifetime, however they continue to be larger than the reference WC-Co, with SH1 being 2.5 times larger and SH2 1.15 times larger. This is probably due to lower porosity and a more homogeneous microstructure of WC-Co.
- Even with a greater dispersion of the lifetime of the cutting edge, the classes NbC-Ni_967 and SH2 had a higher average lifetime, about 1.8 times in relation to the reference WC-Co.

Better performance and lifetime repeatability in a cutting tool are the main goals to be achieved in materials for this purpose. Research will continue to be carried out to obtain a homogeneous microstructure.

Declarations

Confirmation of Authorship

As corresponding author, I Laerte José Fernandes, hereby confirm on behalf of all authors that:

1. The authors have obtained the necessary authority for publication.
2. The paper has not been published previously, that it is not under consideration for publication elsewhere, and that if accepted it will not be published elsewhere in the same form, in English or in

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Figures

Figure 1

Comparison between Niobium carbide hardness and Titanium, Tungsten, Molybdenum and Chromium carbide [3]

Figure 2

Schematic diagram of the pressure-assisted sintering process cycle [17]

Figure 3
Sintering process with dynamic inert gas of the NbC-Ni_967 grade

Figure 4
(a) Example of a dilatometric curve (b) Linear contraction measurement scheme to obtain the dilatometric curve

Figure 5
SH1: Sinter cycle - Sinter-HIP
SH2: Sinter cycle - Sinter-HIP

Figure 6

Example of the force-displacement curve and nanoindentation in the binder.

(a) NbC-Ni_967: 100x

(b) NbC-Ni_967: 200x

(c) WC-Co reference: 100x

(d) WC-Co reference: 200x

(e) SH1: 100x

(f) SH1: 200x

(g) SH2: 100x

(h) SH2: 200x

Micrographs of classes SH, NbC-Ni_967, and WC-Co reference (SEM)

Figure 8

Micrographs of classes SH, and NbC-Ni_967: "Nickel lakes" (OM)

Figure 9

Micrographs of classes SH, and NbC-Ni_967: microstructure heterogeneity and grain size (SEM)

Figure 10

Comparison of average flank wear performance (VB) of class SH cutting tool grades versus reference tool WC-Co and NbC-Ni_967

Figure 11

Lifetime of cutting tools for VB = 0.3 mm: Classes SH, NbC-Ni_967, and WC-Co reference

	NbC-Ni_967	NbC-Ni_SH1	NbC-Ni_967	NbC-Ni_SH1
Stereoscopic Magnifying Glass	(a)	(b)	(c)	(d)
SEM	(e)	(f)	(g)	(h)
	NbC-Ni_967	NbC-Ni_SH2	NbC-Ni_967	NbC-Ni_SH2
Stereoscopic Magnifying Glass	(a)	(b)	(c)	(d)
SEM	(e)	(f)	(g)	(h)
	WC-Co	NbC-Ni_SH1	WC-Co	NbC-Ni_SH1
Stereoscopic Magnifying Glass	(a)	(b)	(c)	(d)
SEM	(e)	(f)	(g)	(h)
	WC-Co	NbC-Ni_SH2	WC-Co	NbC-Ni_SH2
Stereoscopic Magnifying Glass	(a)	(b)	(c)	(d)
SEM	(e)	(f)	(g)	(h)
Wear Mechanism	Abrasion and adhesion	Abrasion and adhesion	Abrasion and adhesion	Abrasion and adhesion

Figure 12

Comparison of wear mechanisms occurred - Abrasion and Adhesion