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ARTICLE

Comparative study of obtaining and characterization of polycrystalline diamond sintered via HPHT with different binders (Nb and Ta)*

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Abstract

Polycrystalline diamond (PCD) is widely used in cutting and drilling tools but faces challenges, including graphitization and environmental and health concerns related to its traditional binders, such as cobalt. This study explores the route to obtaining PCD composites using niobium and tantalum as alternative binders, evaluating composites with 15 wt% binder sintered via HPHT at 1650 °C, 1750 °C, and 1850 °C under 7.7 GPa. The starting powders and milled mixtures were characterized by XRD, particle size analysis, and SEM, while the sintered samples were subjected to densitometry and Brazilian disk compression tests. The samples containing tantalum showed higher densification and compressive strength than those containing niobium, and these properties increased with temperature. Low levels of graphitization were detected, but with negligible impacts on the properties analyzed, indicating these compositions through the HPHT route are promising and more sustainable alternatives to conventional PCD.
Keywords: Polycrystalline diamond, High Pressures and High Temperatures, Niobium, Tantalum.

INTRODUCTION

The development of diamond-based cutting and drilling tools began in the late 19th century, but significant advances were only achieved in the mid-20th century with the advent of the High-Pressure High-Temperature (HPHT) technique. This processing route, still widely used today, enables the production of diamonds with controlled configurations, sizes, and crystalline qualities, making it possible to tailor their properties for specific applications. Among the different variants, polycrystalline diamond (PCD) stands out for its exceptional hardness, high thermal conductivity, and relatively lower cost compared to single-crystal diamond, making it widely used in the cutting and wear tool industry [1].

Despite being one of the hardest known materials, diamond exhibits low fracture toughness, which limits its performance under severe impact and vibration conditions. To overcome this limitation, PCD is typically produced as a composite material, in which diamond particles are bonded by a metallic binder phase. This matrix not only ensures mechanical retention and stress distribution but also influences physicochemical processes at the diamond/metal interface, potentially improving the composite's structural integrity. However, conventional metallic catalysts such as cobalt, iron, and nickel also promote graphitization (the transformation of diamond into thermodynamically stable graphite), which severely compromises the hardness and service life of the tool. Furthermore, cobalt poses environmental and occupational hazards, which have motivated the search for alternative binders [2-5].

In this context, refractory metals such as niobium (Nb) and tantalum (Ta) have emerged as promising substitutes. Recent studies have shown that these elements can form stable carbides during sintering, reducing graphitization and enhancing interfacial adhesion in PCD. Some of these studies have demonstrated that pure Nb and Ta offer competitive performance compared to traditional binders, with additional benefits such as improved oxidation resistance and thermal stability [6-12]. Moreover, the influence of their concentration on the mechanical and thermal properties of PCD has been identified as a key parameter for optimizing

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diamond-based composites.

Therefore, this work investigates the effect of Nb and Ta binders, at controlled mass fractions, on the mechanical properties of polycrystalline diamond composites obtained by HPHT sintering. The objective is to correlate variations in sintering temperature and processing conditions with the modulus of elasticity, densification, and structural performance, contributing to the development of cutting tools with longer tool life and greater stability under extreme conditions.

EXPERIMENTAL PROCEDURES

2.1. Materials and Powder Preparation

Polycrystalline diamond (PCD) powder with 99,9% purity and particle sizes between 40–60 µm (Zhongxiang) was used as the main phase. Niobium (Nb) and tantalum (Ta) powders (99.8% purity, CBMM, Brazil) served as metallic binders. Two mixtures were prepared containing 85 wt% PCD and 15 wt% binder (Nb or Ta). Homogenization was carried out by high-energy planetary ball milling (Pulverisette, Fritsch) using WC-Co jars and balls, at 400 rpm for 20 min, with a ball-to-powder mass ratio of 10:1, in cyclohexane as a milling medium. The slurry obtained was dried in a furnace at 90 °C for 15 min to remove residual solvent. The average particle size for niobium powder was 36,6 µm, and for tantalum 3,48 µm. Despite this difference, the ground mixtures with niobium and tantalum had similar average particle sizes, of 40.03 and 36.03, respectively.

2.2. HPHT Sintering

The milled powders were placed into cylindrical graphite capsules, which were positioned inside CaCO supports. Sintering was performed in a multi-anvil High-Pressure High-Temperature (HPHT) press (DO138B, Ryazantyashpressmash, Russia) at 7.7GPa and three different temperatures: 1650 °C, 1750 °C, and 1850 °C, with a total dwell time of 9 min divided into three cycles of 3 min each. Figure 1 provides a more detailed description of the experimental structure where sintering occurred. More detailed descriptions can be found in previous works by the authors [6-10]. After cooling under pressure to avoid diamond-to-graphite transformation, the samples were cleaned in an ultrasonic bath and chemically etched in a HNO_3:HCl (1:3) solution for 20 min to remove residual graphite.

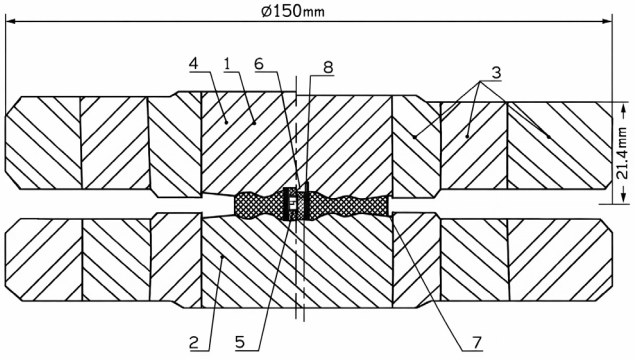

Figure 1: High pressure device used in HPHT treatments. This device is composed of two anvils (1 and 2), supported by the multi-rings (3), and a calcite capsule(4). The PCD is placed into the (5) and the discs (6). During pressing, the compressive gasket (7). The heat is conducted to the sample by a graphite cylinder heater (8).

2.3. Characterization Methods

The starting powders and milled mixtures were characterized by laser diffraction particle size analysis (Cilas 920), scanning electron microscopy with field emission gun (SEM-FEG, Zeiss Auriga 40) coupled with energy dispersive spectroscopy (EDS), and X-ray diffraction (XRD, Rigaku Miniflex II, Cu-Kα radiation, scanning from 20 to 120º (2θ), a step of 0.01º, and speed of 5º/min.). For sintered bodies, density and porosity were determined by the Archimedes method (ASTM B962-13), and Equation A below was used to find the density values.

$$\rho_s = \rho_l \frac{m_s}{m_u - m_i} \quad (A)$$

With ρ_s is the density of the solid sample, ρ_l the liquid density, m_s is the dry mass, m_u = wet mass, and m_i is the immersed mass. Mechanical performance was evaluated through Brazilian disc compressive strength tests (Instron universal testing machine, crosshead speed 1 mm/min). Equation B below was used to find the tensile strength values necessary for calculating Young's modulus.

$$\sigma_t = \frac{2P}{\pi D t} \quad (B)$$

With σt is the tensile strength, P is the maximum load at failure, D is the sample diameter, and t is the sample thickness (height)

RESULTS AND DISCUSSION

3.1. Analysis of starting powders

The particle size distribution analysis revealed average particle sizes of 43.32 µm, 36.60 µm, and 3.48 µm for diamond, niobium, and tantalum powders, respectively. Energy-dispersive X-ray spectroscopy (EDS) confirmed high purity levels (> 99wt.%) for all three starting powders. Figure 2 presents the micrographs of each starting material together with their corresponding X-ray diffraction (XRD) patterns. The results of the X-ray diffraction (XRD) analysis, showing only the characteristic peaks of each element in their respective patterns, are indicative of the purity indicated in the supplier›s reports.

The micrographs presented in Figure 2 reveal a homogeneous particle size distribution for the diamond powder, while the metallic powders exhibit a markedly

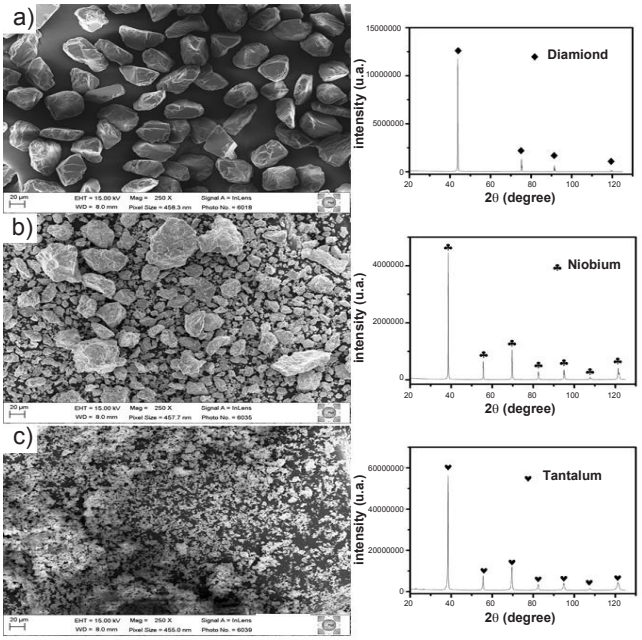

Figure 2: SEM and XRD of starting powders.

heterogeneous distribution. This heterogeneity in metal particle sizes can be advantageous for their function as binders, as the presence of smaller particles enables them to occupy the voids between larger particles. Such packing behavior enhances the densification of the composite, which in turn can improve various mechanical properties.

3.2. Analysis of milled mixtures

The particle size distribution results for the milled mixtures indicated average particle sizes of 40.03 μm and 36.03 μm for the niobium and tantalum containing compositions, respectively. These values suggest that milling at 400 rpm promoted homogenization of the mixtures without inducing significant particle fracture. Figure 2 presents the scanning electron microscopy (SEM) micrographs and X-ray diffraction (XRD) patterns for both mixtures. Analysis of the micrographs confirms that the milling conditions did not generate significant fragmentation.

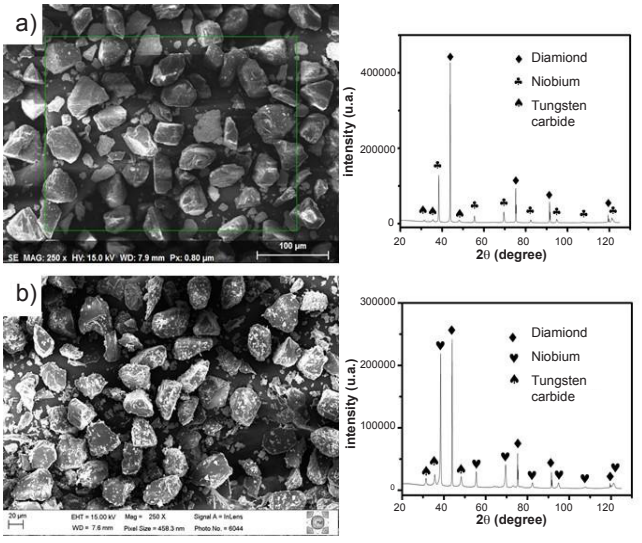

Figure 3: SEM and XRD of milled mixtures

Microstructural analysis also revealed that, although the binder phase coated the surfaces of the diamond particles in both compositions, tantalum exhibited a more pronounced coating effect compared to niobium. This fact may be related to both the nature of the element and the difference in particle sizes between the binder powders, since smaller particles tend to favor coating. In the XRD patterns shown to the right of each micrograph, the diffraction peaks of the metallic binder are observed overlapping with those of diamond, along with the expected tungsten carbide peaks originating from the milling jars and balls.

3.3. Analysis of sintered samples

The SEM micrographs of the diamond composites containing 15 wt% niobium sintered at 1650 °C (a), 1750 °C (b), and 1850 °C (c) reveal significant microstructural changes as the sintering temperature increases, particularly when comparing the 1650 °C sample to those sintered at higher temperatures (shown in Figure 4). The 1650 °C specimen exhibits a more heterogeneous binder distribution, with regions where niobium is concentrated in agglomerates, leaving areas of direct contact between diamond particles and larger niobium islands among the diamond particles. This morphology tends to limit densification and compromise mechanical strength, as the interfacial bonding between particles is less uniform. This observation is supported by the densitometry (Figure 5) and compressive strength (Table I) results, where the highest values for both properties were achieved at the two higher sintering temperatures.

For the specimens sintered at 1750 °C and 1850 °C, a more homogeneous binder distribution across the surface is observed. This redistribution is corroborated by the EDS elemental maps (d–f), where the niobium signal (yellow) appears more evenly dispersed and distributed among the diamond particles (blue), content, and improves phase-to-phase contact. Such behavior is associated with the enhancement of diffusion mechanisms at higher temperatures, which promote binder infiltration and the formation of stronger, more consolidated interfaces. At these elevated temperatures, niobium also forms a more continuous coating over the diamond particles, with more efficient pore filling. The presence of a more interconnected binder network contributes to improved mechanical load transfer between particles and reduces stress concentration sites.

Overall, increasing the sintering temperature improved niobium dispersion and reduced porosity, which directly correlates with the experimentally observed increase in compressive strength (Figure 4 and Table I). EDS mapping confirms that even at concentrations of 15 wt%, niobium progressively coats the diamond particles more uniformly as temperature increases, thereby enhancing the structural integrity of the composite.

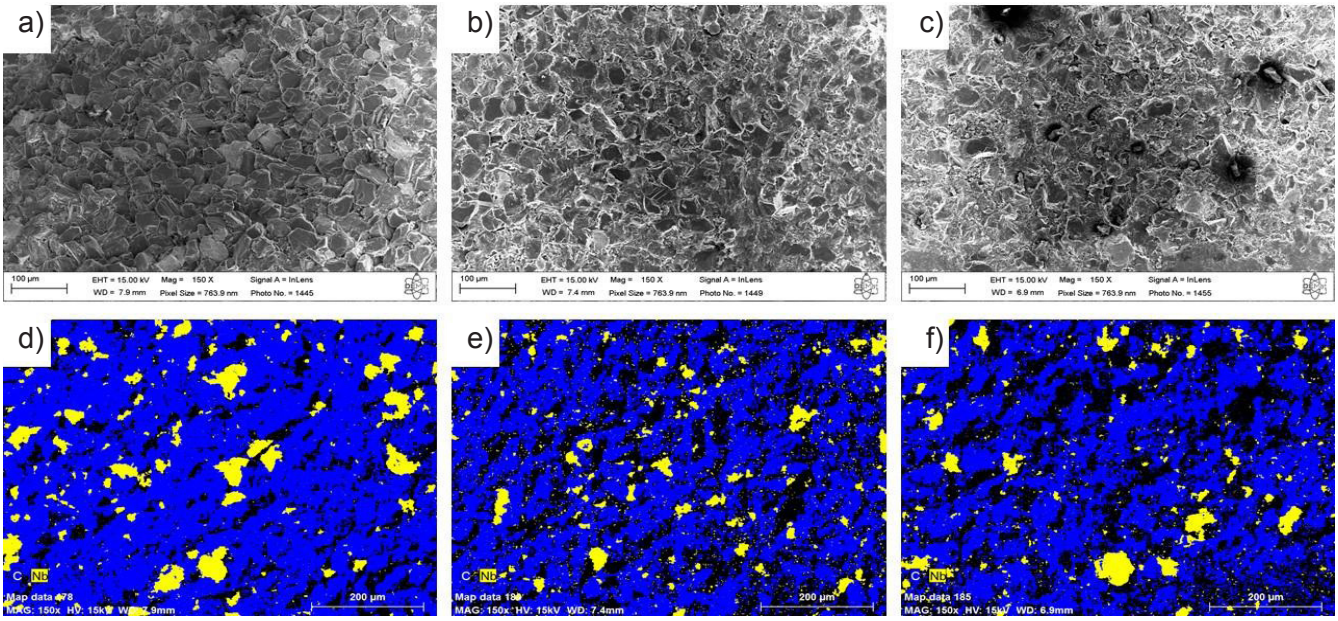

Figure 4: Micrographs of samples sintered with Niobium at 1650 °C (a), 1750 °C (b), and 1850 °C (c), and their respective mappings via EDS.

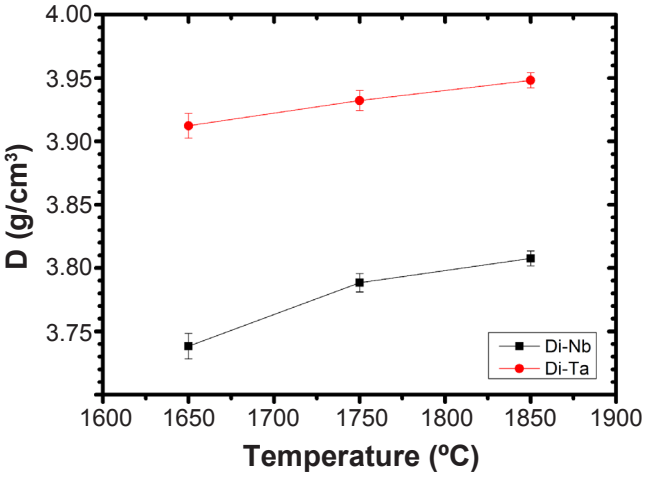

Figure 5: Densimetry of sintered samples.

Table I - Young Modules of sintered samples

Binders	1650 °C	1750 °C	1850 °C
Nb (15 wt%)	1021 ± 19 GPa	1080 ± 17 GPa	1102 ± 14 GPa
Ta (15wt%)	1107 ± 15 GPa	1133 ± 14 GPa	1168 ± 11 GPa

For the composite containing 15 wt% Ta (Figure 6a–c), increasing the sintering temperature also promotes a more uniform coating of the diamond particles; however, the effect is more pronounced than that observed with Nb. The EDS mapping (Figure 6d–f) shows that Ta (in green) displays a finer and more homogeneous distribution even at 1650 °C, with a significant reduction in regions lacking visible binder. This improved dispersion may be related to Ta's higher chemical affinity for carbon and its greater tendency to form stable carbides during sintering.

The more uniform Ta distribution reduces porosity and increases density, contributing to the higher Young's modulus and compressive strength values experimentally observed compared to Nb. In addition to Ta›s greater chemical affinity for carbon, another factor that also favors Ta is its smaller particle size than Nb, since there is a tendency for these properties to improve as the particle size decreases. The presence of a continuous and uniform coating around the diamond particles acts as a barrier to crack propagation, improving the composite's structural integrity. In contrast, although Nb also forms carbides and fills pores as the temperature increases, the presence of agglomerates and partially uncovered regions suggests that load transfer between diamond particles is less efficient, which may explain the lower mechanical values (Figure 5 and Table I).

The densitometry results are consistent with the microstructural analyses, showing an almost gradual progression in densification for Ta and a much more significant increase when comparing the values from 1650 to 1750 than the increase from 1750 °C to 1850 °C in the case of Nb. The combined analysis of densitometry, compression resistance (Young's modulus in Table I), and XRD patterns shows that a low graphitization occurs under the investigated sintering conditions. The progressive increase in apparent density with temperature reflects densification and improved particle bonding, conditions that are typically compromised when graphite formation is significant. This densification trend is accompanied by increases in compressive strength and Young›s modulus, suggesting the preservation of the diamond structure and the establishment of efficient charge transfer between particles. Furthermore, the XRD results

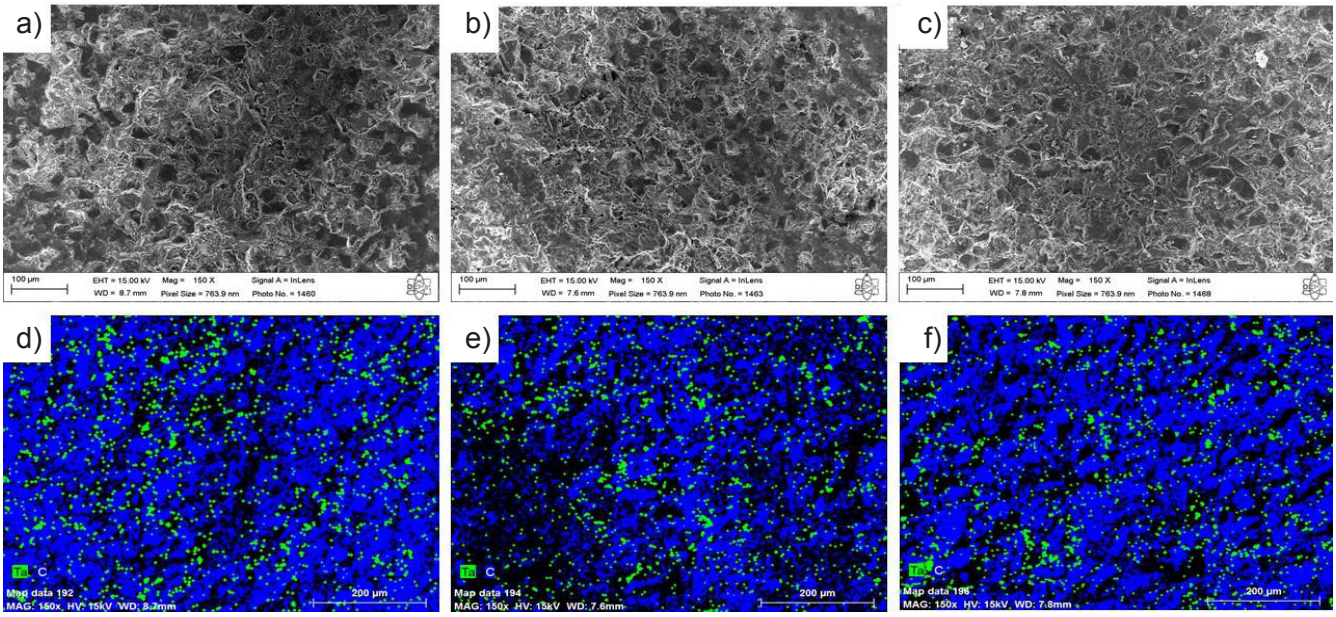

Figure 6: Micrographs of samples sintered with Tantalum at 1650 °C (a), 1750 °C (b), and 1850 °C (c), and their respective mappings via EDS.

(Figure 6) revealed small graphite peaks which may be partly attributed to mold contamination, but since the diffraction patterns show a dominance of diamond peaks and carbide phases (NbC or TaC), it can be inferred, along with the results of the other analyses, that both the presence of graphite and WC contamination during milling were negligible to the point of not significantly interfering with the properties.

The low graphite-related degradation pathways and the formation of carbides from the binders explains the consistent improvement in mechanical performance with increasing temperature, since higher temperatures favor the formation reactions of these new phases, and thus the Ta-C and Nb-C bonds reduce the amount of free carbon available to form graphitic structures, reinforcing the conclusion that the Nb and Ta binders effectively stabilized the diamond phase throughout the sintering process.

XRD results confirmed the presence of NbC and TaC for each composition at all sintering temperatures, along with small peaks corresponding to contaminants such as WC and graphite. In the case of graphite peaks, this trend may be correlated with contamination from the graphite mold tooling used during sintering. Despite meticulous cleaning efforts after sintering, the contamination could not be completely avoided or removed, leaving traces of graphite in the intergranular regions. The addition of a binder results in the formation of new phases. But the sintering conditions were insufficient to melt Nb or Ta, so graphite formation may be related to voids between diamond or binder particles.

Reported Young's modulus values for sintered PCD systems, including Co-bonded and binderless compacts, typically range from ~850 to 1150 GPa depending on binder type and characterization method [13-15]. The values obtained in this study (1021–1168 GPa) are consistent with the upper range reported for dense HPHT-processed diamond compacts.

CONCLUSIONS

• The results demonstrate that both Nb and Ta are promising alternative binders for PCDs, effectively preserving the diamond phase during sintering.

• Increasing the sintering temperature promoted improved binder dispersion and enhanced interfacial consolidation, which directly contributed to the progressive increase in density, Young's modulus, and compressive strength.

• Ta exhibited superior performance across all evaluated temperatures, achieving more uniform coverage of diamond particles and acting more effectively as a crack propagation barrier. This can be explained by its greater chemical affinity for carbon, and also by the fact that it was used in a smaller average particle size than niobium

• EDS mapping confirmed a progressively homogeneous coating for both binders, while XRD analysis revealed the presence of NbC and TaC phases without significant graphite peaks

• The combination of high densification, enhanced mechanical performance, and the low graphitization signals indicates that the sintering process preserved the structural integrity of the diamond phase in both compositions, with Ta powder showing a more pronounced effect.

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DATA AVAILABILITY

The data that support the findings of this study are available from the corresponding author, upon reasonable request.

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