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# Towards the Optimization of Microstructure and Mechanical Properties of NbC-based Cermets

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***Abstract*** **–** The NbC-Ni cermets have shown potential as an eco-friendly alternative to WC-Co cemented carbide tools. However, their room temperature hardness and flexural strength necessitate further improvement. Various techniques, such as grain inhibition and reinforcement, have been employed to enhance these properties. This study explores the effects of adding 4% VC and 4% $Mo_2C$ on the microstructure and mechanical properties of NbC-12Ni cermets. The NbC-12Ni cermets were produced using vacuum liquid phase sintering (LPS) at temperatures of 1400°C and 1450°C. The incorporation of 4% VC and 4% $Mo_2C$ significantly enhanced the properties of NbC-Ni cermets. SEM analysis indicated substantial grain refinement as a result of grain inhibition mechanisms. The grain sizes observed ranged from 6.84 µm in NbC-12Ni to 2.34 µm in the sample containing 4 wt. % VC and 4 wt. % $Mo_2C$, sintered at 1450°C. At this temperature, the incorporation of VC and $Mo_2C$ resulted in a notable 21.5% increase in average hardness, despite a slight decrease in fracture toughness from 10.224 MPa√m to 9.324 MPa√m. Additionally, the flexural strength improved, reaching a maximum of 936.39 N/mm for NbC-Ni4VC4$Mo_2C$, marking a 14.1% increase compared to the NbC-Ni cermet.

*Keywords – Niobium Carbide, Cermets, Microstructure, Grain inhibition, Mechanical Properties*

## I. INTRODUCTION

Tungsten carbide-cobalt (WC-Co) cemented carbides are renowned for their outstanding mechanical properties, but they suffer from high solubility in iron-based alloys at elevated temperatures, which accelerates tool wear due to adhesion with Fe[1]–[3]. Additionally, both tungsten (W) and cobalt (Co) are classified as critical raw materials (CRMs) by the European Union and are considered high-risk due to their limited availability. In 2013, cobalt was classified as a carcinogen by the European Commission (through REACH) and the U.S. National Toxicology Program (NTP), prompting industries to seek safer alternatives[4]–[6].

Niobium carbide (NbC) emerged as a promising substitute, known for its high melting point of 3522°C, excellent wear resistance, and hardness of 19.6 GPa. These properties make NbC a strong candidate for cutting tool applications and biomedical uses such as bone implants, where it enhances tribocorrosion resistance in titanium alloys[7]–[9]. NbC-Ni cermets, among various binding agents like Co[10], [11], Fe[12], [13], Ni[14], [15], and high entropy alloys[16], [17], have gained attention as potential replacements for WC-Co cemented carbides due to their favorable mechanical attributes and lower density.

While NbC-Ni cermets do not match the thermal conductivity, fracture toughness, and flexural strength of WC-Co, they exhibited superior wear resistance and chemical stability against iron and steel.[2], [18]. However, previous studies have noted significant carbide particle growth during the sintering of NbC and Ni alone, indicating areas for further improvement[18],[19].

Research efforts are increasingly focused on enhancing the mechanical attributes of NbC-Ni cermets, particularly through the addition of secondary carbides. Previous studies using vacuum pressureless sintering have shown that additives like VC, $Mo_2C$, and TiC can significantly reduce NbC grain size, thereby improving hardness and fracture toughness. $Mo_2C$ addition has been found to increase hardness but decrease toughness[20], [21]. Similarly, introducing TiC into NbC-12%Ni has demonstrated its potential to inhibit grain growth, resulting in enhanced hardness and toughness through vacuum liquid phase sintering. Various studies have examined the combined effects of additives like ($Mo_2C$ + TiC) [22], and (WC + TiC), aiming to balance hardness and toughness[23],[24].

This study investigated the combined effect of VC and $Mo_2C$ additions, along with sintering temperature, on grain growth inhibition and the mechanical properties of NbC-Ni cermets. The composition included 4 wt. % VC and 4 wt. % $Mo_2C$ added to NbC-Ni cermets. The powders were processed through vacuum liquid phase sintering at two different temperatures. The findings provided valuable information on the synergistic effects of VC and $Mo_2C$ on NbC-Ni cermets, with implications for their application in various industrial settings.

## II.MATERIALS AND METHOD

### A. *Materials preparation*

NbC (FSSS-1.10µm), VC (FSSS-2.60µm), $Mo_2C$ (FSSS-1.90µm) sourced from Huarui Metals, and Ni (FSSS-5µm) from Merck were utilized to prepare NbC-Ni based cermets with additional reinforcements. The initial powder morphology is shown in **Fig. 1**, and their chemical composition is detailed in **Table 1**. Precise weighing of the powders was done using an analytical balance to match the specified compositions. These weighed powders were placed into a high-density polyethylene (HPDE) vessel with a 0.25L mill volume and mixed at 200 RPM using cylindrical WC-6Co balls (3mm and 5mm diameter). The mixing took place in an ethanol medium for 24 hours in a low-energy ball mill (WIseMixBML-2), with ball: powder of 10:1. After mixing, the slurry was vacuum dried at 50°C for 5 hours in a controlled environment. The dried powders were then compacted into circular (11mm diameter) and rectangular (25mm x 5mm x 2mm) shapes using uniaxial pressing at 200MPa, followed by cold isostatic pressing at 250MPa. The compacts underwent sintering with a heating rate of 10°C/min up to 1200°C and 5°C/min up to 1400°C and 1450°C, with an isothermal dwell time of 1 hour in a dynamic vacuum environment maintained at $10^{-2}$ Pascal. After sintering, the samples were slowly cooled inside the furnace. Temperature measurements were conducted with a thermocouple up to 1000°C and a 2-beam laser pyrometer up to 1400°C-1450°C. The sintered cermets were then ground using various SiC grit papers and polished with 6µm, 3µm, and 1µm diamond paste.

### B. *Characterization*

The characterization of the sintered specimens involved multiple techniques. Using Archimedes' principle, the density of the sintered samples was assessed. SEM imaging of polished sintered samples was executed in secondary electron (SE) mode on a JEOL JSM6490A SEM with energy-dispersive X-ray Spectrometer (EDX) capability. The line intercept technique was employed to measure the average grain size. Hardness measurements (HV30) were carried out using a hardness tester (Model HVRVU-187.5) with a 30Kgf indentation load and a dwell time of 15 seconds. Palmqvist indentation toughness (KIC) was determined using the method described by Shetty et al.[25], based on the radial crack lengths around the Vickers indentation. The reported values were the averages of three readings.

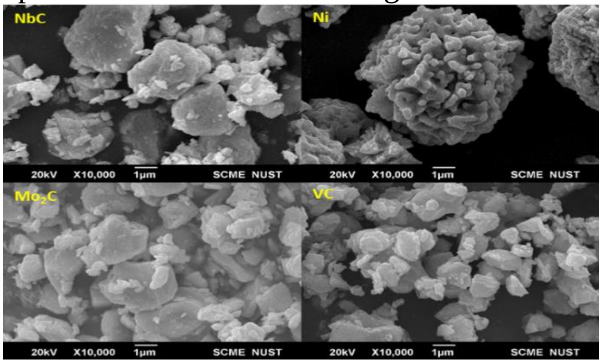


Fig. 1 Morphology of Initial powder used in the fabricatin of cermets

Table 1. Chemical Composition of fabricated cermets

| **Sample Vol%** | **Abbreviation** | **Sample composition Wt.%** | | | |
|---|---|---|---|---|---|
| | | NbC | Ni | VC | $Mo_2C$ |
| **Density(g/cm$^{3}$)** | ρ | 7.83 | 8.90 | 5.77 | 9.18 |
| **NbC12Ni** | NbC12Ni | 86.57 | 13.43 | --- | --- |
| **NbC12Ni4VC4Mo$_2$C** | 4VC4Mo$_2$C | 79.02 | 13.43 | 2.91 | 4.64 |

For assessing flexural strength, a standard 3-point bend test was conducted using a Shimadzu AGX-Plus Universal Testing Machine equipped with a 20kN load cell. The test utilized a span length of 16mm and a crosshead displacement rate of 0.1mm/min.

## III. RESULTS

The density measurements of the fabricated samples, as depicted in **Fig. 2**, indicated a clear temperature dependence. The relative density (R.D.) of both samples was higher at 1450°C, with the 4VC4Mo$_2$C sample exhibiting a slightly higher R.D. compared to the NbC-12Ni sample.

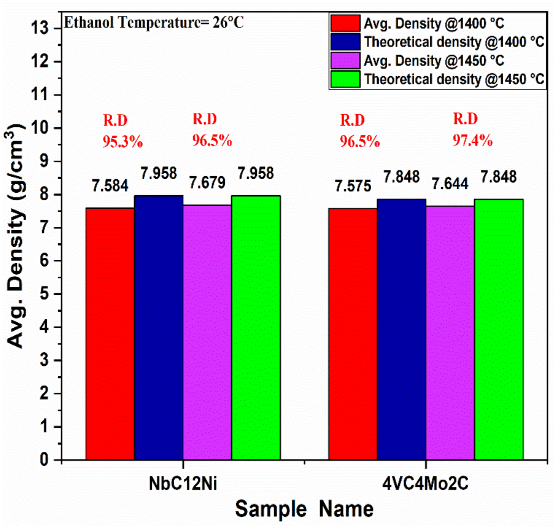


Fig. 2 Density of the fabricated Samples

SEM micrographs in **Fig. 3** and **Fig. 5** revealed numerous micro-pores in both samples at 1400°C, whereas at 1450°C, the presence of micro-pores was significantly reduced, suggesting enhanced densification at the higher temperature. Elemental distribution maps, obtained through EDX analysis and depicted in **Fig. 4**, confirmed the distribution of Ni in the binder regions and NbC in the carbide regions for NbC-12Ni cermet.

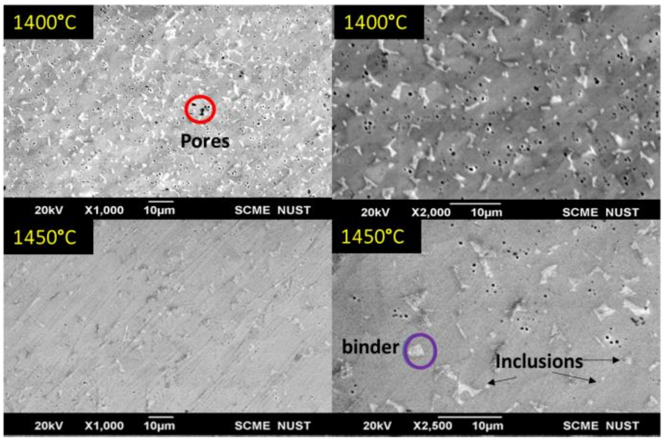


Fig. 3 SE-SEM image of NbC-12Ni cermet sintered at 1400°C- 1450°C

The elemental distribution in **Fig. 4**, demonstrated the presence of V and Mo within both the NbC grains and the Ni binder, with a uniform distribution of the binder phase observed in the micrographs.

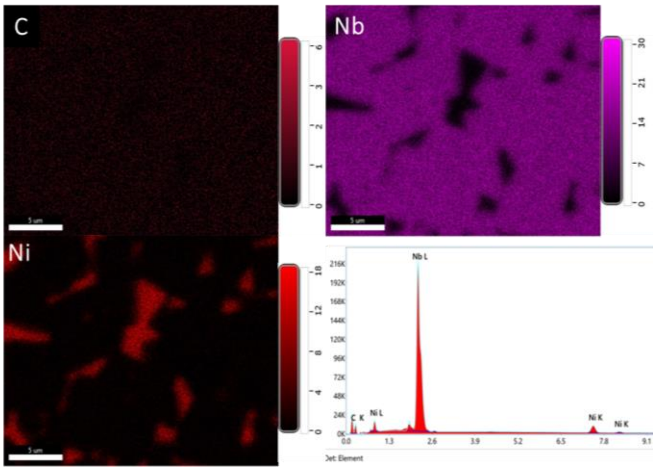


Fig. 4 : EDX distribution of Ni,C and Nb in NbC12Ni cermet vol.%

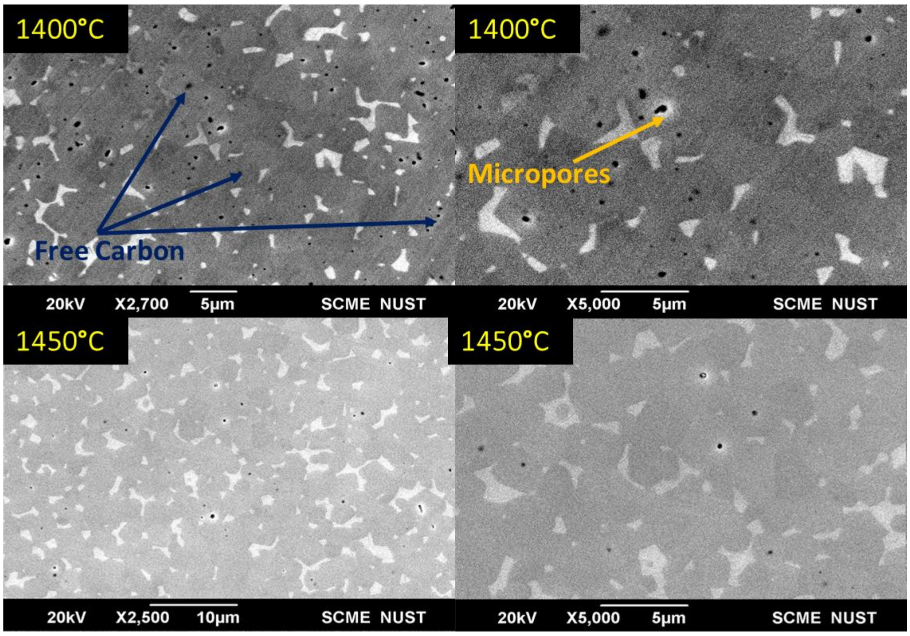


Fig. 5 : SE-SEM image of $NbC12Ni4VC4Mo_2C$ cermets sintered at 1400°C & 1450°C

**Table 2**. illustrates the avg. grain size of the fabricated samples, highlighting significant grain refinement with the addition of $4VC4Mo_2C$.

Hardness testing results, shown in Hata! Başvuru kaynağı bulunamadı., indicated an improvement in hardness with increasing temperature for both cermets. Notably, the addition of $4VC4Mo_2C$ at 1450°C resulted in an approximate 21.5% increase in hardness. Fracture toughness results, depicted in. **Fig. 8** followed a similar trend for temperature. However, the addition of $4VC4Mo_2C$ slightly compromised fracture toughness, which decreased from 10.224 $MPa.m^{1/2}$ to 9.324 $MPa.m^{1/2}$.

The flexural strength of the fabricated cermets exhibited an increasing trend with temperature as depicted in **Fig. 9**. The maximum flexural strength of 936.39 N/mm was achieved for NbC-Ni-$4VC4Mo_2C$, representing a 14.1% increase compared to the base cermet. The comparative analysis of hardness and fracture toughness, as depicted in **Fig. 9** , demonstrated that the material exhibited a favorable combination of hardness and fracture toughness at 1450°C.

Table 2. Grain Size of fabricated samples

| Sample | Avg. Grain size (μm) @1400°C | Avg. Grain size (μm)@1450°C |
|---|---|---|
| NbC12Ni | 4.34 | 6.84 |
| $4VC4Mo_2C$ | 3.19 | 2.34 |

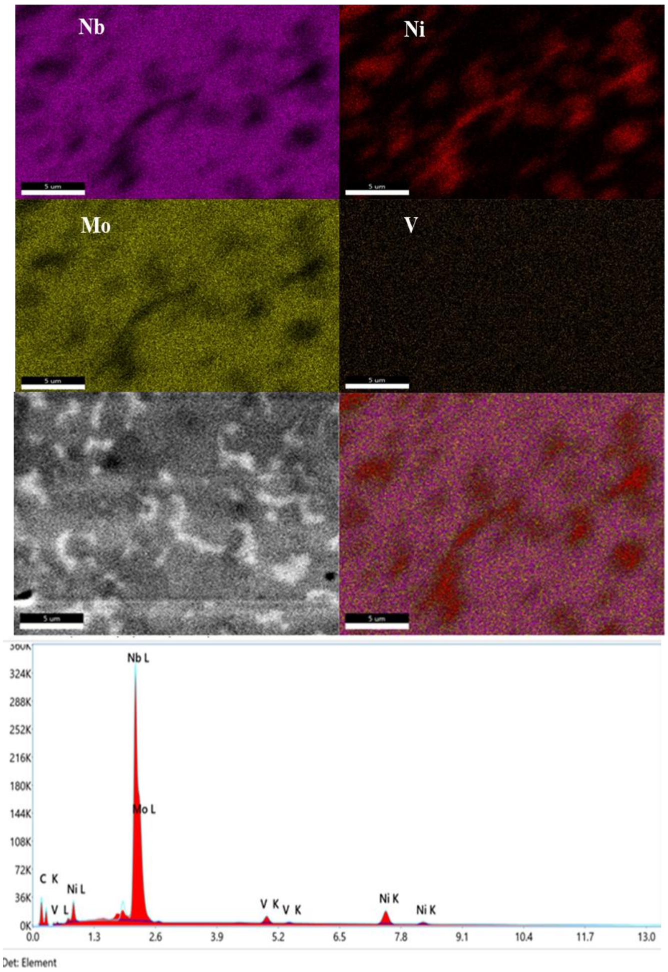


Fig. 6 EDX Map of elemental distribution for NbC12Ni4VC4$Mo_2C$ vol.%

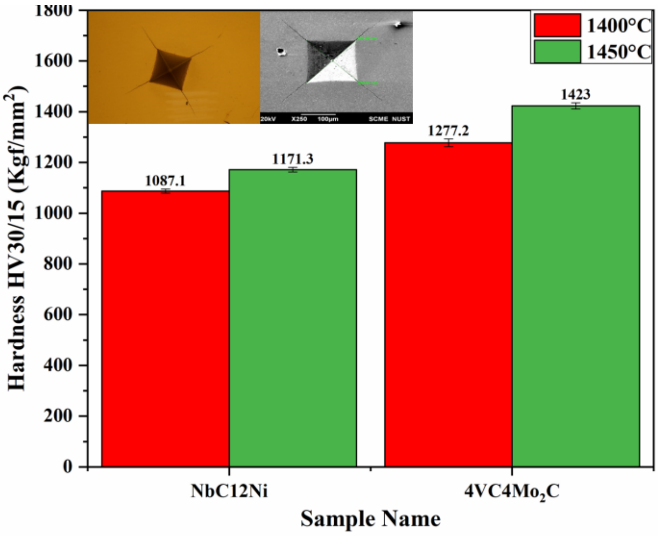


Fig. 7 Vickers Hardness (HV30) of the fabricated Samples

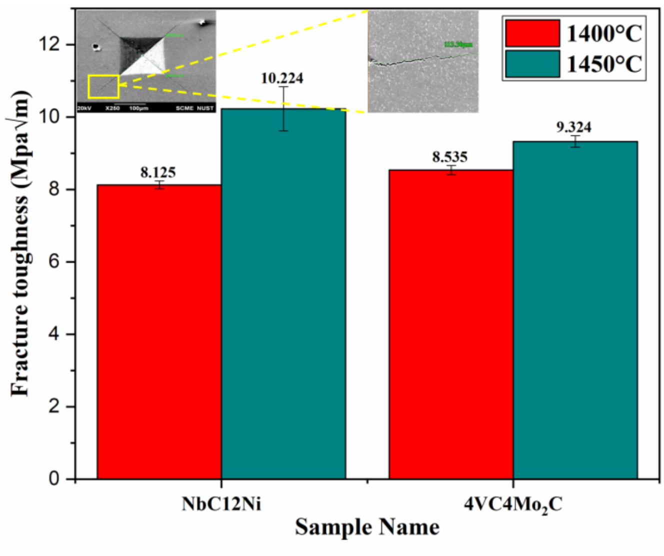


Fig. 8 Fracture Toughness of fabricated samples

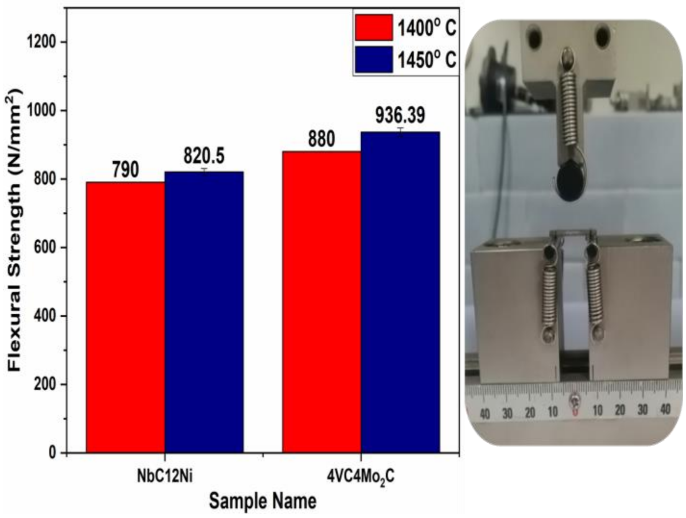


Fig. 9 Flexural Strength of fabricated samples

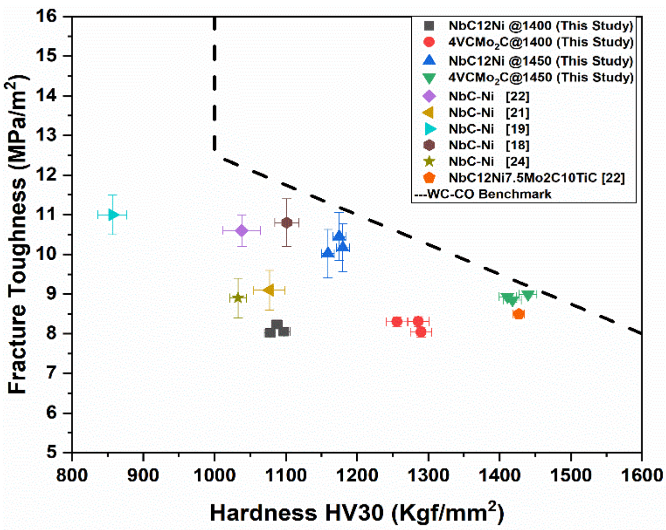


Fig. 10 Comparative Analysis of HV30 Vs Fracture Toughness with Literature

This performance was observed to be near the WC-Co benchmark values found in the literature.

## IV. DISCUSSION

The results highlight the substantial influence of temperature and secondary carbides on the densification and mechanical properties of NbC-Ni-based cermets. Higher sintering temperatures, particularly 1450°C, enhanced relative density (R.D.), as fewer micro-pores were observed, consistent with established theories on pore elimination and grain boundary diffusion. The uniform distribution of Ni and NbC phases, along with V and Mo incorporation, suggests effective mixing and sintering, crucial for homogenous mechanical properties.

The addition of 4VC4$Mo_2C$ led to notable grain refinement, inhibiting grain growth during sintering, aligning with previous studies on grain growth inhibitors [20], [26]. This refinement resulted in a 21.5% increase in hardness at 1450°C, following the Hall-Petch relationship, though fracture toughness slightly decreased, reflecting a common trade-off in cermets.

Flexural strength improvements, particularly the 14.1% increase for NbC-Ni-4VC4$Mo_2C$ cermets, underscore enhanced load-bearing capacity and resistance to bending stresses, essential for applications requiring high structural integrity.

## V. CONCLUSION

1. Sintering at 1450°C significantly improved the relative density (R.D.) of NbC-12Ni and NbC-4VC-4$Mo_2C$ cermets, with the latter showing slightly higher R.D. Reduced micro-pores at this temperature indicate enhanced densification, crucial for mechanical robustness.
2. The inclusion of 4VC4$Mo_2C$ effectively suppressed grain growth, leading to significant grain refinement and enhancing the hardness and mechanical properties of the cermets.
3. The hardness of the cermets increased by 21.5% at 1450°C with the addition of 4VC4$Mo_2C$. However, this improvement was accompanied by a slight reduction in fracture toughness, reflecting a common trade-off in cermet materials.
4. The flexural strength of the cermets increased with temperature, peaking at 936.39 N/mm for NbC-Ni-4VC4$Mo_2C$, marking a 14.1% improvement over NbC-Ni cermets. This underscores enhanced load-bearing capacity and mechanical stress resistance.

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