مجلة الدراسات التربوية والعلمية - كلية التربية - الجامعة العراقية
العدد السابع والعشرون - المجلد الحادي عشر - علوم الفيزياء - آيار 2026 م
doi.org/10.52866/esj.2026.11.27.12

Effect of Vanadium Addition on Microstructure and Mechanical Properties of Nickel Alloys Prepared with Powder Metal Technology

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Abstract :

This study focuses on the fabrication and characterization of nickel–vanadium alloys prepared by the powder metallurgy technique, aiming to investigate the influence of different vanadium contents (0–20 wt.%) on the crystal structure, microstructure, mechanical properties, and mechanical wear behavior at 500 °C. X-ray diffraction (XRD) analysis confirmed that nickel preserved its face-centered cubic (FCC) structure in all compositions, while vanadium maintained its body-centered cubic (BCC) structure, indicating the stable coexistence of both phases without the formation of unwanted phase transformations. The structural analysis revealed that increasing the vanadium content led to a noticeable reduction in the crystallite size of the nickel matrix, highlighting the effectiveness of vanadium in restricting grain growth and refining the microstructure. These observations were further supported by scanning electron microscopy (SEM) results, which showed a gradual decrease in grain size from approximately 120 nm for pure nickel to about 96 nm with the addition of 20 wt.% vanadium. This behavior demonstrates the role of vanadium as an efficient grain refiner by stabilizing grain boundaries and limiting their growth at elevated temperatures. The refined microstructure had a direct and positive impact on the mechanical properties of the alloys. The Vickers hardness values increased steadily from 148.6 HV for pure nickel to 178.2 HV at the highest vanadium content, which can be attributed to the increased grain boundary density and enhanced resistance to dislocation movement. In addition, compressive strength measurements showed a significant improvement with increasing vanadium content, rising from 420 MPa to 512 MPa. This enhancement confirms the strong correlation between grain size refinement and the improved ability of the alloy to withstand plastic deformation under applied loads.The mechanical wear behavior of the alloys was also evaluated, and the results demonstrated a continuous reduction in the wear rate with increasing vanadium content. This improvement is mainly attributed to the increased hardness and the formation of a fine and homogeneous microstructure, which effectively reduces material removal during sliding contact. Regarding true porosity, a slight increase was observed with higher vanadium additions; however, all porosity values remained below 3%, indicating good densification and acceptable structural integrity of the prepared samples.

Keywords:Nickel alloys, Vanadium addition, Powder metallurgy, Microstructural refinement, Mechanical properties, Wear behavior.

تأثير إضافة الفاناديوم على البنية المجهرية والخواص الميكانيكية لسبائك النيكل المحضرة بتقنية ميتالورجيا المساحيق

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مستخلص:

تم في هذه الدراسة تحضير سبائك النيكل–فاناديوم باستخدام تقنية تعدين المساحيق، ودراسة تأثير إضافة نسب مختلفة من الفاناديوم (0–20 wt.٪) على الخواص البنيوية، والمجهرية، والميكانيكية، وسلوك البِلى الميكانيكي عند درجة حرارة 500 C°. أظهرت نتائج حيود الأشعة السينية (XRD) أن النيكل حافظ على بنيته المكعبة متمركزة الوجوه (FCC)، بينما احتفظ الفاناديوم ببنيته المكعبة متمركزة الجسم (BCC) في جميع التراكيب، مما يدل على استقرار الطورين وعدم حدوث تحولات طورية غير مرغوب فيها.بينت النتائج أن زيادة محتوى الفاناديوم أدت إلى انخفاض ملحوظ في الحجم البلوري وحجم الحبيبات، حيث انخفض حجم الحبيبات من حوالي 120 nm للنيكل النقي إلى نحو 96 nm عند إضافة 20 wt..٪ فاناديوم، مما يعكس الدور الفعال للفاناديوم في تنعيم البنية المجهرية وتقييد نمو الحبيبات. وقد انعكس هذا التحسين البنيوي بشكل مباشر على الخواص الميكانيكية، إذ ارتفعت قيم صلادة فيكرز من 148.6 HV إلى 178.2 HV، كما تحسنت مقاومة الانضغاط من 420 MPa إلى 512 MPa مع زيادة نسبة الفاناديوم.كما أظهرت نتائج البِلى الميكانيكي انخفاضا مستمرا في معدل البِلى مع زيادة محتوى الفاناديوم، ويُعزى ذلك إلى زيادة الصلادة وتكون بنية دقيقة ومتجانسة تقلل من فقدان المادة أثناء التلامس الميكانيكي. وعلى الرغم من ملاحظة زيادة طفيفة في المسامية الحقيقية مع زيادة الفاناديوم، إلا أن قيم المسامية بقيت ضمن حدود منخفضة (<3٪)، مما يدل على تحقق تكاثف جيد وتماسك بنيوي مقبول للعينات المحضرة.

الكلمات المفتاحية: سبائك النيكل، إضافة الفاناديوم، تعدين المساحيق، تنعيم البنية المجهرية، الخواص الميكانيكية، سلوك البِلى الميكانيكي.

1. Introduction

Nickel alloys are an advanced engineering material that is of wide interest in harsh industrial applications, such as aerospace, power generation, and petrochemicals, due to their outstanding mechanical and thermal properties, as well as their good resistance to corrosion and oxidation. However, exposure of these alloys to extreme operating environments, especially at high temperatures, leads to the deterioration of their surface properties and the acceleration of corrosion processes, which negatively affects their service life and efficiency [1].

Corrosion at high temperatures is one of the most complex problems, as multiple factors such as the nature of the oxidizing medium, the microstructure of the alloy, and its chemical composition interfere. For this reason, recent research has tended to improve the corrosion resistance of nickel alloys through the deliberate addition of different alloying elements, which contribute to the formation of protective and stable oxide layers on the surface [2,3].

Vandium is one of the alloys that has shown promise in this area, which has the ability to improve the mechanical and surface properties of metal alloys. Vandium contributes to the purification of the microstructure, enhances granular bonding, and contributes to the formation of protective oxides that reduce the rate of oxygen diffusion and corrosive agents [4,5].

Previous studies have shown that the addition of calculated ratios of vanadium to nickel alloys can lead to a marked improvement in corrosion resistance, especially in environments with variable temperatures. This improvement is due to changing surface reaction mechanisms, forming more cohesive oxide layers and thermal stability [6,7].

The effect of vandium is not limited to a single temperature, but varies according to the thermal field, which necessitates a systematic study of the behavior of vandium-containing nickel alloys at multiple temperatures, to understand the relationship between chemical composition, corrosion mechanism, and long-term performance [8,9].

Based on the above, this study aims to analyze the effect of vanadium addition on the corrosion resistance of nickel alloys at different temperatures, by evaluating wear rates, studying the microstructure, and analyzing oxidation products, thus contributing to the development of alloys with improved performance for advanced industrial applications [10–12].

2- Raw materials (powders)

Nickel powder was used in this study.**(Ni)**The base material and vanadium(V) powder were used as a reinforcing element. Particle size was verified using standard sieves to ensure compliance with experimental requirements. Table (1) shows the basic properties of the powders used.

Table (1): Powders used in the study

T	Powder type	purity(%)	Particle size(µm)	country of origin	density of the stack(g/cm³)
1	Ni	99.9	≤ 63	Germany	8.9
2	V	99.6	≤ 63	England	6.11

3- Preparing raw materials

Preparation of Raw Materials

The vanadium powder was dried in an oven at a certain temperature.**200 °C**The mixture was left to stand for two hours to remove moisture. Afterward, the powders were prepared according to specific volumetric ratios due to the density difference between nickel and vanadium, ensuring a homogeneous mixture. Weighing was performed using a high-precision Sartorius electronic balance with an accuracy of 0.0001 g.

4- Mixing and grinding powders

Mixing and Milling of Powders

A quantity of**3 g**The powder was ground in a homemade mill using 10 mm diameter steel balls at a 5:1 weight ratio for 1–2 hours at 500 rpm, with the aim of achieving homogeneous distribution and reducing particle size. Table (3-3) shows the parameters of the grinding process.

Table (2):
Powder grinding parameters

teacher	Value
Type of mill	Homemade
grinding time	hours 2–1
Grinding speed	rpm 500
Ball diameter	mm 10
Balls to powder ratio	5:1

5- Pressing stage

The powders were compressed using a single-axis compression method with a hardened steel die, where a pressure of700 MPaFor one minute. The procedure was performed using a UTM-HOYTOM device with a power of 170 kN. Cylindrical samples with approximate dimensions of 10 mm in diameter and 5 mm in height were produced, suitable for subsequent examinations.

6- Vickers Hardness Test

The Vickers hardness test was carried out according to the following steps to evaluate the resistance of the prepared samples to plastic deformation:

1. The surface of the specimen was first prepared by grinding and polishing to obtain a smooth and defect-free surface.
2. The specimen was then placed firmly on the stage of the hardness testing machine.
3. A specified load (F) was applied using a diamond pyramid indenter with an angle of 136° between opposite faces for a fixed dwell time.
4. After removing the load, the two diagonals of the indentation (d_1 and d_2) were measured using the optical microscope attached to the device.
5. The average diagonal length (d) was calculated using the following relation:

$$d = \frac{d_1 + d_2}{2} \ldots\ldots (1)$$

6. The Vickers hardness value (HV) was then calculated using the equation:

$$HV = \frac{1.854\,F}{d^2} \ldots\ldots (2)$$

7. where F is the applied load in kgf and d is the average diagonal length in mm.
8. The measurements were repeated at several different locations on the specimen surface, and the average hardness value was reported to ensure accuracy and repeatability.

7- Wear Test

The mechanical wear test was conducted according to the following steps to evaluate the wear behavior of the prepared samples under sliding conditions:

1. The surface of each specimen was cleaned and polished to ensure uniform contact during the test.
2. The specimen was weighed accurately before the test using a digital balance, and the initial weight (W_1) was recorded.
3. The specimen was then fixed in the wear testing machine, and a specified normal load was applied.
4. The test was performed under controlled conditions of sliding speed and sliding distance for a predetermined time.
5. After completing the test, the specimen was removed, cleaned to eliminate wear debris, and weighed again to record the final weight (W_2).
6. The weight loss (ΔW) was calculated using the relation:

$$\Delta W = W_1 - W_2 \dots\dots (3)$$

7. The wear rate was then determined based on the weight loss and test parameters.
8. The test was repeated for all samples under the same conditions to ensure reliable and comparable results.

8- X-ray Diffraction (XRD) Analysis

X-ray diffraction (XRD) technique was employed to investigate the phase composition and crystal structure of the prepared samples. The diffraction patterns were used to identify the crystalline phases and evaluate the structural characteristics of the materials. In addition, the average crystallite size was estimated from the broadening of the diffraction peaks using the Debye–Scherrer equation, which relates the peak width to the size of coherently diffracting domains.

The crystallite size was calculated using the following Debye–Scherrer equation:

$$D = \frac{K\lambda}{\beta \cos\theta} \dots\dots (4)$$

where D is the average crystallite size, is the shape factor (typically taken as 0.9), λ is the X-ray wavelength, β is the full width at half maximum (FWHM) of the diffraction peak expressed in radians, θ and is the Bragg

diffraction angle.

9- Results and Discussion

9-1 X-ray diffractionXRD

X-ray diffraction results for the pure nickel sample showed thatAt 500 °C, there are three major peaks at levels (111), (200), and (220), which are due to the cubic structure and face-centeredness (FCC) of nickel. These peaks were characterized by their clarity and relative sharpness, reflecting good crystal regularity and relatively high crystal volumes, indicating the stability of the main phase of pure nickel. With the addition of 5% vanadium, the nickel peaks continued to appear at the same positions, while new peaks emerged at levels (110) and (200), belonging to the (BCC) structure of vanadium. This indicates that vanadium retained its original crystal phase within the alloy, without altering the crystal phase of nickel. This was accompanied by a significant decrease in the calculated crystal volume of nickel, suggesting that the presence of vanadium atoms within the structure inhibited crystal growth and prevented free expansion, resulting in a reduction in crystal volume. When the vanadium content was increased to 10%, the vanadium peaks became more pronounced compared to the 5% sample, while the nickel peaks continued to appear with a further decrease in crystal volume values. This behavior demonstrates that increasing the vanadium content promotes crystallinity. Its phase increases its effect in reducing the size of nickel crystals. [13]

In the sample containing 15% vanadium, the peaks of both components persisted.Ni and V were clearly visible. A variation in the calculated crystalline values was observed, with some levels, such as (220) for nickel, showing a relatively higher crystalline size compared to the other peaks. This can be explained by local rearrangement or directed growth in certain crystalline directions, without negating the overall effect of reducing crystalline size due to the addition of vanadium. At 20% vanadium, the characteristic vanadium peaks became more pronounced and crystalline, while the nickel peaks continued to appear with smaller crystalline sizes than the pure sample. This indicates that the two original phases

(FCC) Ni and (BCC) V clearly coexist within the alloy, and that increasing vanadium leads to a further reduction in the crystalline size of nickel, reflecting vanadium's role in limiting crystal growth and maintaining fine-grained structures. [14]

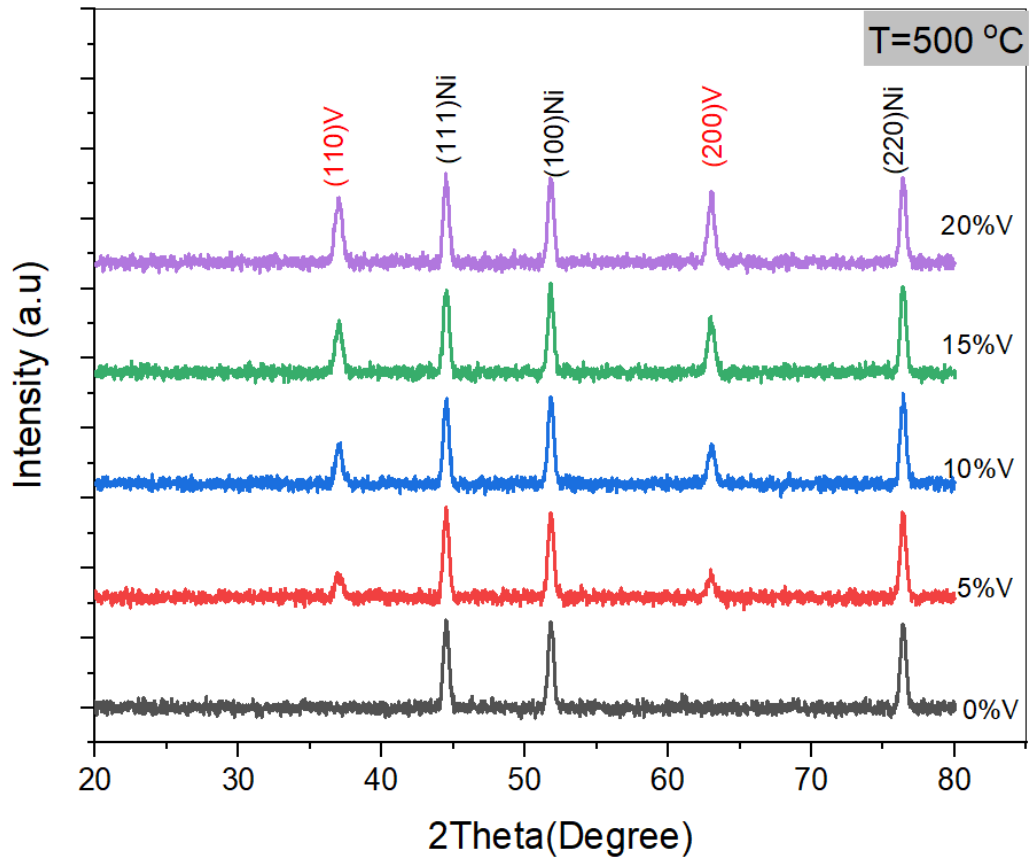

Figure (1) X-ray diffractionXRD

Table (3) X-ray diffraction resultsXRD

	2θ (deg)	FWHM (deg)	d-spacing (Å)	Size Gs (nm)	hkl	Material	Cell Type	Crystal System
Ni Pure	44.49	0.089	2.0346	111.2	(111)	Ni	FCC	Cubic
	51.85	0.115	1.7620	88.7	(200)	Ni	FCC	Cubic
	76.38	0.099	1.2459	117.1	(220)	Ni	FCC	Cubic
V 5%	42.14	0.086	2.1426	114.3	(110)	V	BCC	Cubic
	44.44	0.127	2.0369	77.9	(111)	Ni	FCC	Cubic
	51.78	0.087	1.7640	116.8	(200)	Ni	FCC	Cubic
	61.12	0.126	1.5150	84.1	(200)	V	BCC	Cubic
	76.28	0.097	1.2473	119.7	(220)	Ni	FCC	Cubic
V 10%	42.14	0.121	2.1426	81.0	(110)	V	BCC	Cubic
	44.39	0.124	2.0392	79.7	(111)	Ni	FCC	Cubic
	51.72	0.093	1.7660	109.2	(200)	Ni	FCC	Cubic
	61.12	0.108	1.5150	98.4	(200)	V	BCC	Cubic
	76.17	0.113	1.2488	102.7	(220)	Ni	FCC	Cubic
V 15%	42.14	0.125	2.1426	78.4	(110)	V	BCC	Cubic
	44.34	0.106	2.0415	92.7	(111)	Ni	FCC	Cubic
	51.66	0.092	1.7680	110.2	(200)	Ni	FCC	Cubic
	61.12	0.125	1.5150	84.9	(200)	V	BCC	Cubic
	76.07	0.085	1.2502	137.0	(220)	Ni	FCC	Cubic
V 20%	42.14	0.116	2.1426	84.2	(110)	V	BCC	Cubic
	44.28	0.112	2.0438	88.3	(111)	Ni	FCC	Cubic
	51.60	0.097	1.7700	104.6	(200)	Ni	FCC	Cubic
	61.12	0.125	1.5150	85.0	(200)	V	BCC	Cubic
	75.97	0.097	1.2516	118.9	(220)	Ni	FCC	Cubic

9-2 Scanning Electron MicroscopeSEM

Scanning electron microscope images showed(SEM) At a heat treatment of 500°C, the pure nickel sample had an average grain size of about 120.34 nm, which reflects a relatively crystalline structure with well-defined grain boundaries. With the addition of 5% vanadium, the grain size decreased to

115.27 nm, indicating that the vanadium atoms began to inhibit grain growth by stabilizing their boundaries and reducing the atomic diffusion rate at this temperature. When the vanadium was increased to 10%, the decrease in grain size continued to reach 110.85 nm, where the distribution became more homogeneous compared to the pure sample. This illustrates the increasing role of vanadium as a grain refiner by limiting the growth of large grains. In the sample containing 15% vanadium, the grain size reached 101.42 nm, reflecting an additional enhancement in the structural smoothing process[19], as the grains appeared more regular and had a higher boundary density, which helps to increase hardness and wear resistance. In the sample with 20% vanadium, the grain size recorded its lowest value at 500°C at about 96.38 nm. This gradual decrease in grain size with increasing vanadium content confirms that vanadium effectively limits grain growth at this temperature. The SEM results show that the addition of vanadium to nickel leads to a significant smoothing of the grain structure, as the grain size gradually decreases with increasing vanadium content. This decrease is consistent with what was observed by X-ray diffraction (XRD)[14].

Table (4) ResultsSEM

Vanadium ratio(V%)	Granular size(nm)
0	120.34
5	115.27
10	110.85
15	101.42
20	96.38

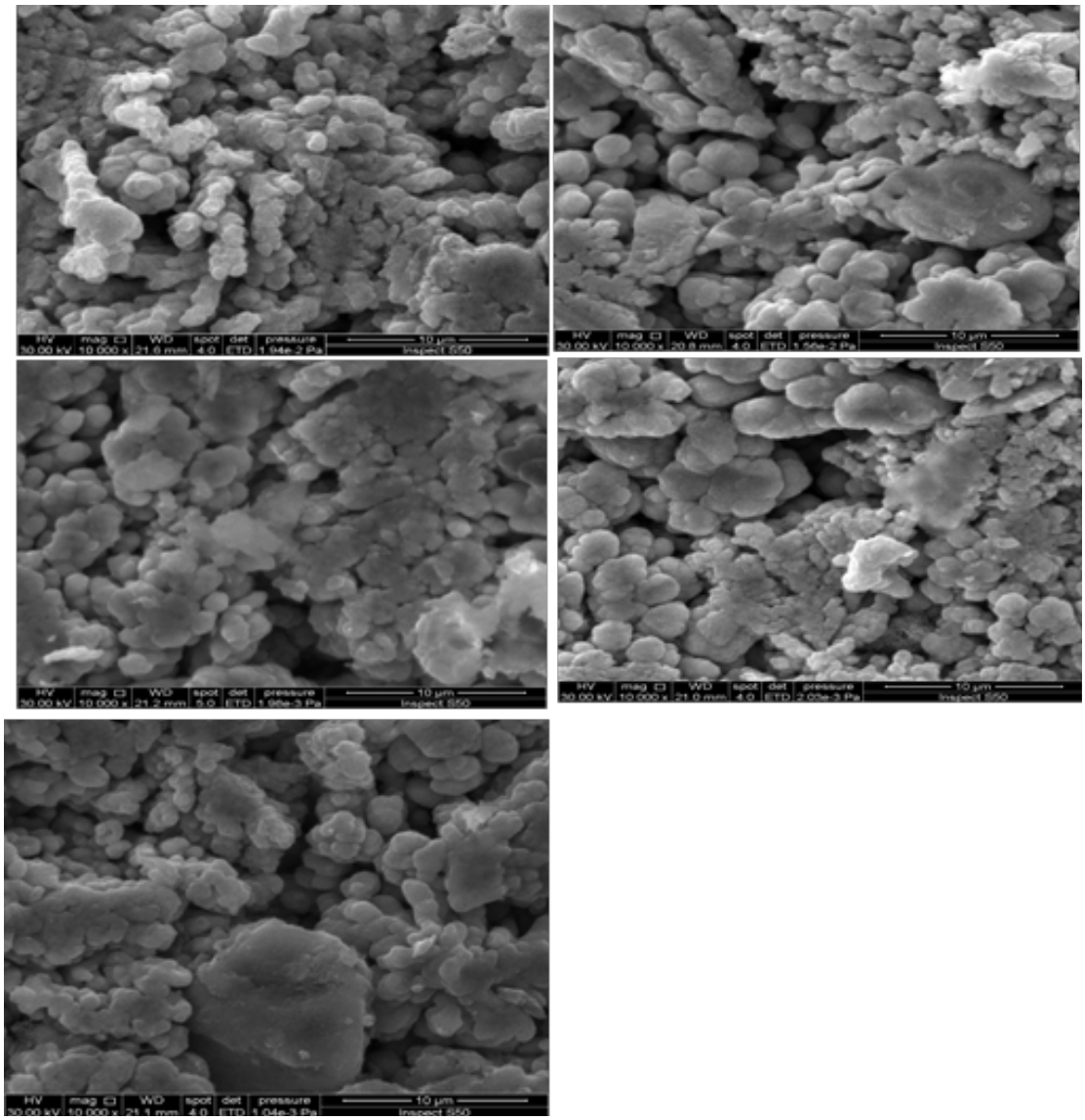

Figure (2) Scanning electron microscope images

9-3 Vickers hardness

Vickers hardness measurements of the heat-treated samples showed thatAt 500 °C, the pure nickel sample exhibited an average hardness value of approximately 148.6 HV, consistent with the larger granular structure observed by scanning electron microscopy (SEM). This structure reflects a limited resistance to deformation due to the relatively large grains and low grain boundary density.

With the addition of vanadium, a gradual increase in hardness was observed, with values rising to156.3 HV at 5% V, then to 163.9 HV at 10% V.

This improvement is attributed to the inhibitory effect of vanadium on grain growth, which leads to a reduction in grain size and an increase in grain boundary density, which enhances resistance to dislocation movement and leads to increased hardness.[15]

At higher vanadium concentrations (15% and 20%), the increase in hardness continued, reaching171.4 HV and 178.2 HV respectively. This indicates that vanadium acts effectively as an alloying modifier, contributing to the formation of fine grain boundaries that effectively inhibit dislocations, thus increasing the material's resistance to plastic deformation.

The results of hardness at500 °C confirms that the addition of vanadium leads to a clear increase in the hardness of nickel alloys. This improvement is directly related to what was observed through XRD and SEM of a gradual decrease in crystalline and grain size. Therefore, controlling the microstructure by adding vanadium at this temperature is an important factor in improving the mechanical properties of the alloys.[45]

Table (5) Vickers Hardness(HV)

Vanadium ratio(V%)	hardness(HV)
0	148.6
5	156.3
10	163.9
15	171.4
20	178.2

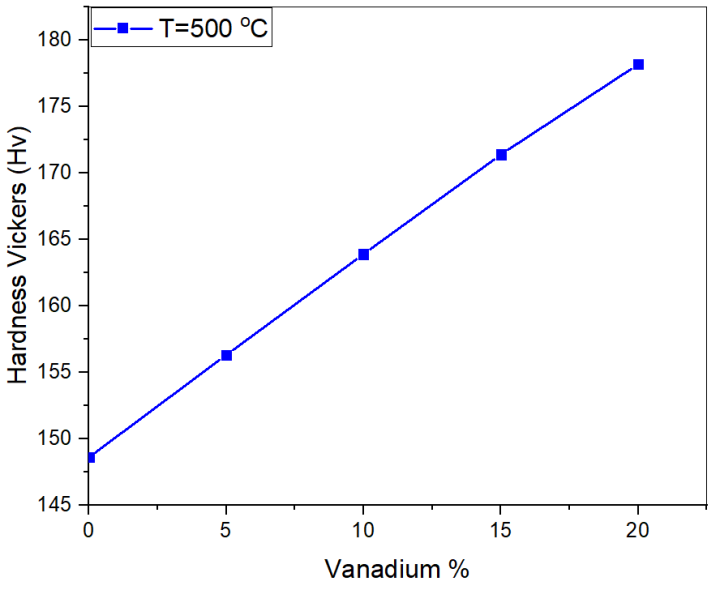

Figure (3) Vickers hardness

6-4 Compressive Strength

The results of the compressive strength test for the heat-treated samples showed thatAt 500°C, the pure nickel sample exhibited an average compressive strength of approximately 420 MPa. This value reflects the relatively larger grain size observed by scanning electron microscopy (SEM), which reached about 120.34 nm. This indicates a lower grain boundary density and reduced ability to inhibit dislocations, resulting in relatively limited compressive strength. With the addition of vanadium, a gradual increase in compressive strength was observed, rising to 445 MPa at 5% V, then to 468 MPa at 10% V, reaching 490 MPa at 15% V, and finally reaching a maximum value of 512 MPa at 20% V. This clear improvement reflects the structural role of vanadium, as it stabilizes the grain boundaries and reduces their growth rate, leading to a smoother structure and a reduction in grain size. The direct relationship between the reduction in grain size and the increase in compressive strength is that reducing the grain dimensions increases the density of the grain boundaries, and these boundaries act as barriers to the movement of dislocations, thus increasing the alloy's ability to resist deformation under compressive load.[16]

The results show whenAt 500°C, the addition of vanadium resulted in a marked improvement in the compressive strength of nickel alloys. This improvement is directly related to observations made by both XRD (reduction in crystal size) and SEM (smoothing of the grain structure), as well as the observed increase in Vickers hardness. This confirms that controlling the microstructure through vanadium addition is an effective method for enhancing the mechanical properties of alloys at this temperature.

Table (6) Compressive Strength

Vanadium ratio (V%)	Compressive strength (MPa)
0	420
5	445
10	468
15	490
20	512

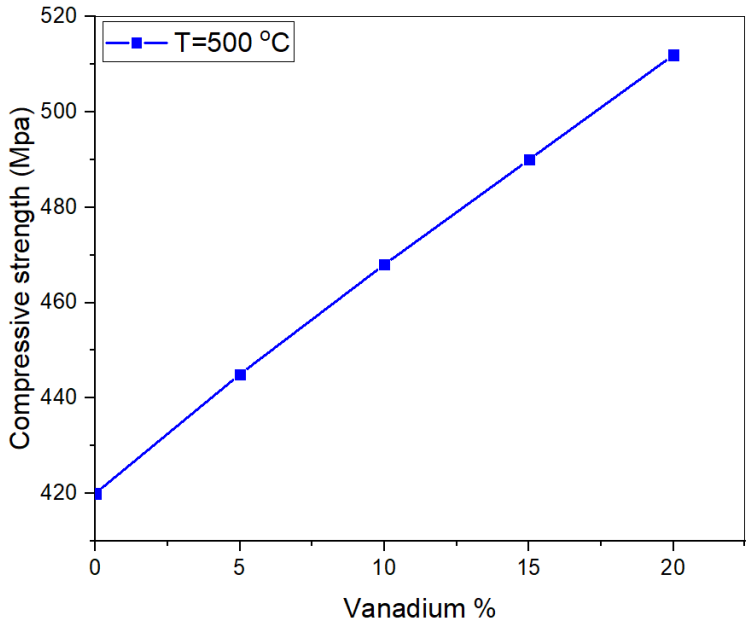

Figure (4) Compressive strength

9-5 Wear rate

The wear test results for the pure nickel sample showed thatAt 500°C, the wear rate reached approximately 4.85×10^{-6} g/cm², a relatively high rate reflecting the limited resistance of pure nickel to friction and surface wear. This can be explained by the high rate being related to the relatively large grain size and low hardness, which allows for easier plastic deformation during friction and faster material loss from the surface. With the addition of vanadium, the wear rate gradually decreased to 4.32×10^{-6} g/cm² at 5% V, then 3.89×10^{-6} g/cm² at 10% V. This decrease reflects the role of vanadium in reducing the grain size and increasing the density of the grain boundaries, which increases hardness and reduces the susceptibility of the surface to dislocation and wear during the friction process.[16]

At higher percentages (15% and 20%), the wear rate continued to decrease, reachingThe values of 3.55×10^{-6} g/cm² and 3.28×10^{-6} g/cm², respectively, indicate that increased vanadium significantly improves wear resistance. This is attributed to enhanced mechanical protection through increased hardness and the formation of a more homogeneous fine-grained structure, as well as vanadium's po-

tential contribution to the formation of more stable surface layers that resist mechanical wear.

The results of wear and tear upon-The 500°C test shows that the addition of vanadium reduces the wear rate and improves mechanical corrosion resistance, which is consistent with what was observed through hardness and compressive strength tests, and confirms the direct relationship between the fine microstructure and the improvement of the mechanical and surface behavior of the alloys.

Table (7) Wear rate at500 °C

Vanadium ratio (V%)	Wear rate (×10⁻⁶ g/cm)
0	4.85
5	4.32
10	3.89
15	3.55
20	3.28

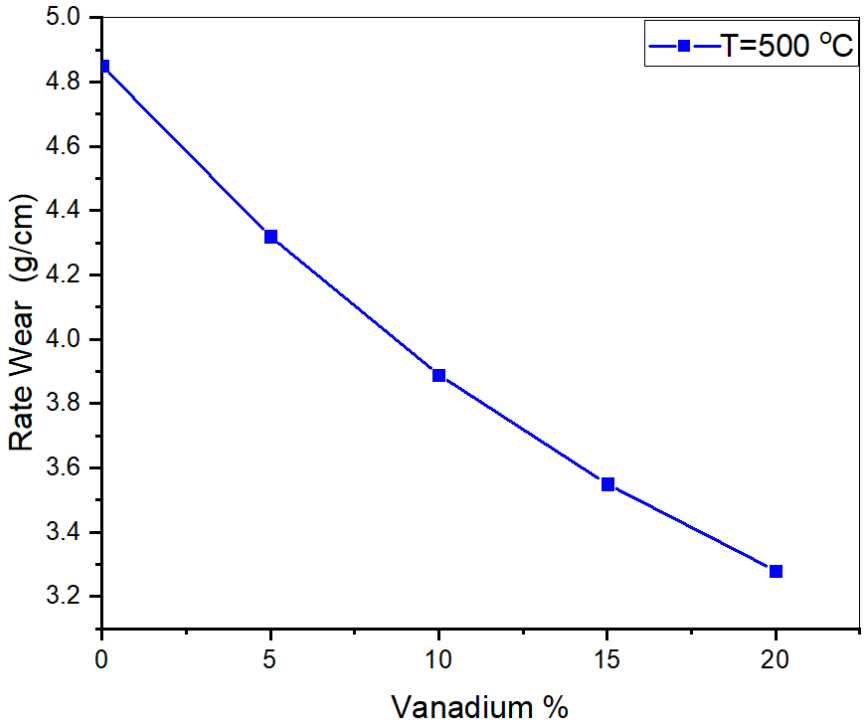

Figure (5) Wear rate

9-6 True porosity

The true porosity results of the heat-treated samples showed thatAt 500°C, the pure nickel sample had a porosity of approximately 2.3%, a percentage indicating that the sintering process at this temperature was insufficient to close all interfacial spaces,

leaving some open pores within the structure. With the addition of vanadium, the values gradually increased to 2.5% at 5% V and 2.6% at 10% V, reaching 2.9% at 20% V. This slight increase in porosity with increasing vanadium content can be explained by the nature of vanadium itself, which has a lower density than nickel and affects the sintering mechanism, somewhat hindering the complete convergence of grains. However, the values remained within a range (less than 3%), indicating that the microstructure was relatively cohesive despite the persistence of some pores. Thus, it can be said that porosity at 500°C is one of the indicators that the sintering did not reach its optimal level and that raising the temperature is necessary to reduce it and improve the structural homogeneity of the alloy.[17]

Table (8) True Porosity

Vanadium ratio(V%)	True porosity(%)
0	2.3
5	2.5
10	2.6
15	2.8
20	2.9

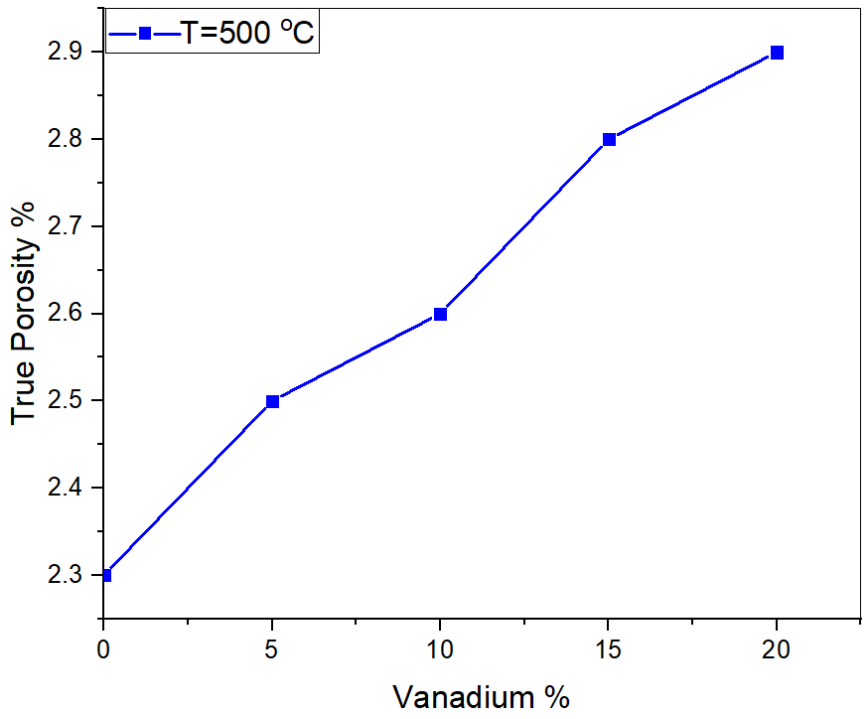

Figure (6) True porosity

Conclusions

The results of the study showed that the addition of vanadium to nickel alloys resulted in a clear smoothing of the crystal and granular structure while maintaining the stability of the two primary phases. The reduction in granular size also contributed to improved mechanical properties, as Vickers' hardness and compressive strength increased with increased vanadium content. In addition, the wear rate was significantly reduced due to improved hardness and structural homogeneity. These results confirm that vanadium is an effective ingredient for improving the performance of nickel alloys at high temperatures.

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