

ТВЕРДЫЕ РАСТВОРЫ НА ОСНОВЕ
ОКСИДОВ МОЛИБДЕНА – ВАНАДИЯ, ВЫПОЛНЯЮЩИЕ РОЛЬ
УПРАВЛЯЕМЫХ РЕДОКС-КАТАЛИЗАТОРОВ ДЛЯ ВЫРАЩИВАНИЯ
НИКЕЛЬМАТРИЧНЫХ КОМПОЗИТОВТ. В. СВИРИДОВА¹⁾, А. С. ЛОГВИНОВИЧ¹⁾, Д. В. СВИРИДОВ¹⁾¹⁾Белорусский государственный университет, пр. Независимости, 4, 220030, г. Минск, Беларусь

Аннотация. Изучены структура и морфология электрохимически осаждаемых металломатричных композитов, представляющих собой пленки гальванического никеля с инкорпорированными редокс-активными оксидными частицами $\text{MoO}_3 : \text{V}_2\text{O}_5$. Выполненное электронно-микроскопическое исследование электроосажденных композитных пленок и фрактальный анализ атомно-силовых изображений свидетельствуют о том, что эффективность зародышеобразования при осаждении матричного металла на частицах $\text{MoO}_3 : \text{V}_2\text{O}_5$ определяется редокс-активностью оксидной фазы. Это обстоятельство открывает возможность для получения мелкокристаллического металлического осадка из плотноупакованных кристаллитов никеля. Сформированные таким образом пленки композита $\text{Ni} - 0,6\text{MoO}_3 : 0,4\text{V}_2\text{O}_5$ характеризуются повышенной коррозионной стабильностью и износостойкостью по сравнению с гальваническим никелем.

Ключевые слова: металломатричный композит; смешанный оксид молибдена – ванадия; зародышеобразование; фрактальный размер; коррозионная стабильность; износостойкость.

Благодарность. Работа выполнена при финансовой поддержке Министерства образования Республики Беларусь в рамках государственной программы научных исследований «Химические процессы и технологии» на 2026–2030 гг. (задание 2.1.05.2 «Химически управляемые капсулирующие покрытия на основе неорганических полимеров», № гос. регистрации 20261628).

Образец цитирования:

Свиридова ТВ, Логвинович АС, Свиридов ДВ. Твердые растворы на основе оксидов молибдена – ванадия, выполняющие роль управляемых редокс-катализаторов для выращивания никельматричных композитов. *Журнал Белорусского государственного университета. Химия.* 2026;1:61–67 (на англ.). EDN: ZZWCQX

For citation:

Sviridova TV, Logvinovich AS, Sviridov DV. Molybdenum – vanadium oxide solid solutions used as a tunable redox catalyst for electrochemical growing of nickel-matrix composites. *Journal of the Belarusian State University. Chemistry.* 2026;1:61–67. EDN: ZZWCQX

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MOLYBDENUM – VANADIUM OXIDE SOLID SOLUTIONS USED AS A TUNABLE REDOX CATALYST FOR ELECTROCHEMICAL GROWING OF NICKEL-MATRIX COMPOSITES

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Abstract. The structure and morphology of electrochemically-derived metal-matrix composite obtained by embedment of redox-active $\text{MoO}_3 : \text{V}_2\text{O}_5$ oxide particles in the galvanic nickel was investigated. The electron microscopic studies of electrochemically grown composite films and fractal analysis of atomic-force microscopic images provide an evidence that the efficiency of nucleation of matrix metal on the $\text{MoO}_3 : \text{V}_2\text{O}_5$ particles is dependent on its redox activity yielding fine closely packed crystallites. The resultant composite film $\text{Ni} - 0.6\text{MoO}_3 : 0.4\text{V}_2\text{O}_5$ film demonstrates radically improved (as compared to bare galvanic nickel) wear-resistant and corrosion resistant properties.

Keywords: metal-matrix composite; molybdenum – vanadium mixed oxide; nucleation; fractal dimension; corrosion stability; wear resistance.

Acknowledgements. This work was carried out with the financial support of the Ministry of Education of the Republic of Belarus within the framework of the state programme of scientific research «Chemical processes and technologies» for 2026–2030 (assignment 2.1.05.2 «Chemically-controlled encapsulating coatings based on inorganic polymers», state registration No. 20261628).

Introduction

The metal-matrix composites consisting from the matrix metal with the embedded oxide [1; 2], boride [3], and carbide [4] particles or industrial diamonds [5] has attracted much attention in the last decades due to the possibility of integration of advantages of codeposited materials. In particular, this permits one to combine plasticity of the matrix metal with the microhardness inherent in the embedded inorganic phase [6; 7]. As the result, the metal-matrix composites, which can be readily deposited onto the metal substrates exhibiting complex surface shape, show promise as the materials for wear-resistant and antifriction functional coatings [5; 8]. Moreover, in many cases the reinforcement of metal deposit through embedment of the particles of the second phase is accompanied with the enhancement of metal matrix chemical stability [6; 8; 9]. It has been shown previously that the process of composite electrodeposition can be enhanced by using redox-active inorganic compounds (e. g. molybdenum, vanadium, and tungsten oxides) as the second phase as the result of localisation of metal crystallisation directly at the surface of the embedded particles [10; 11]. The effect of redox-active particles on the metal matrix growing should offer fresh possibilities in exerting control over the structure of metal-matrix composites permitting one to change the size and orientation of crystallites in the matrix metal. Additional opportunities in changing the structure and morphology of metal-matrix composite grown through redox mechanism could arise in the case of mixed oxides representing substitutional solid solutions that can be obtained via polycondensation of oxo-molybdate and oxo-vanadate oligomers under solvothermal conditions [12]. In these molybdenum – vanadium oxide solid solutions the replacement of vanadium ions in the V_2O_5 lattice with molybdenum ions leads to the partial reduction of V(V) ions into V(IV) state [12] that enhances the redox activity of the resultant mixed oxide.

Here we demonstrate the possibility of controlling the process of the electrochemical formation of nickel-matrix composites using $\text{MoO}_3 : \text{V}_2\text{O}_5$ oxide solid solutions that behave as the dispersed phase with the tunable redox activity.

Materials and methods

The molybdenum – vanadium mixed oxides of variable composition $[\text{xMoO}_3 : (1 - \text{x})\text{V}_2\text{O}_5]$ have been prepared via thermoinduced polycondensation of oxo-compounds in aqueous medium [13]. For this purpose, the molybdenum – vanadium mixed oxyacid obtained from the $(\text{NH}_4)_6\text{Mo}_7\text{O}_{24} + \text{Na}_2\text{VO}_4$ aqueous solution employing ion exchange resin technique was incubated at 100 °C for 4 h [12]. The composition of the resultant mixed oxide was controlled by changing the mole ratio of molybdate and vanadate precursors. The synthesised oxide particles were separated from the reaction solution by centrifugation and dried at room temperature.

The electrolyte used for deposition of nickel-matrix composites ($\text{Ni} - \text{MoO}_3 : \text{V}_2\text{O}_5$) was a Watts-type bath [14] containing 200 g/L NiSO_4 , 50 g/L NiCl_2 , 35 g/L H_3BO_3 . The molybdenum – vanadium mixed oxide particles were added to the electrolyte under ultrasonic agitation in the amount 0.5 g/L immediately before the composite

film deposition. The deposition was performed in a static electrolyte (i. e. under convective mass-transport conditions) on to the steel substrates at the current density 2 A/dm^2 .

Infrared (IR) spectra were recorded employing IRTracer-100 spectrometer (*Shimadzu*, Japan). The scanning electron microscopy (SEM) studies of $\text{MoO}_3 : \text{V}_2\text{O}_5$ particles and $\text{Ni} - \text{MoO}_3 : \text{V}_2\text{O}_5$ composite films were performed with Leo-1420 electron microscope (*Carl Zeiss*, Germany). Surface morphology of composite films was investigated employing atomic force microscopy (AFM) with the use of TT-2 microscope (*AFM Workshop*, USA). The fractal analysis of AFM images was carried out with the use of *Gwiddion* software. For evaluation the fractal dimension the cube counting method was used [15; 16]. The X-band electron paramagnetic resonance (EPR) spectra were recorded at 77 K using EMX-8 spectrometer (*Bruker*, USA). The concentration of paramagnetic centres in the molybdenum – vanadium mixed oxides was evaluated by double integration of EPR spectrum using a $\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$ crystal with known number of spins as a reference standard. Wear resistance of the coatings was tested under dry sliding conditions at a load of 1.1 kg/cm^2 . The corrosion properties of $\text{Ni} - \text{MoO}_3 : \text{V}_2\text{O}_5$ were investigated in comparison with bare galvanic nickel in 5 mol/L NaCl employing electrochemical impedance spectroscopy with PGSTAT101 potentiostat (*Metrohm Autolab*, the Netherlands). All the potentials were measured against the Ag/AgCl , Cl^- (saturated) electrode.

Results and discussion

The simultaneous polycondensation of molybdenum and vanadium oxyacids leads to the substitution of some positions in the cationic sublattice of V_2O_5 by molybdenum ions yielding $x\text{MoO}_3 : (1-x)\text{V}_2\text{O}_5$ solid solution that manifests itself in the IR spectrum (fig. 1) as the addition absorption band at $830\text{--}850 \text{ cm}^{-1}$ corresponding to the vibrations of $-\text{Mo}-\text{O}-\text{V}-$ group [17] (this band arises between the band at 755 cm^{-1} corresponding to the vibrations of $-\text{V}-\text{O}-\text{V}-$ group [18] and that at 899 cm^{-1} corresponding to the vibrations of $-\text{Mo}-\text{O}-\text{Mo}-$ group [19]).

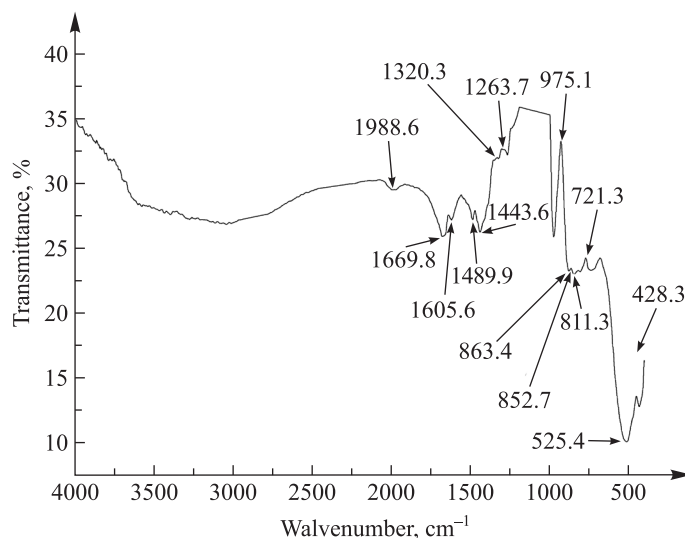


Fig. 1. IR spectrum of $x\text{MoO}_3 : (1-x)\text{V}_2\text{O}_5$ mixed oxide ($x = 0.6$)

The SEM images shown in fig. 2 provide an evidence that the morphology $x\text{MoO}_3 : (1-x)\text{V}_2\text{O}_5$ particles grown through the polycondensation route varies with the molybdenum-to-vanadium mole ratio in the mixed oxyacids used as the precursors. It may be suggested that the inclusion of molybdenum ions into the V_2O_5 lamellae suppresses their lateral growth but facilitate lamellae binding. As the result, contrastingly to the micron-scale whiskers observed at low MoO_3 loading ($x = 0.25$), the compact regular microcrystals of mixed oxide are formed $x = 0.4$ (cf. fig. 2, *a* and *b*); further increase in the molybdenum content leads to the phase segregation and growing of needle-like MoO_3 crystallites, which are formed together with prismatic crystallites of molybdenum – vanadium oxide solid solution (see fig. 2, *c*). The observed evolution of habitus of microcrystallites forming the mixed oxide phase (including the release of bare hexagonal MoO_3 at large loading of molybdenum oxide) agrees well with the results of X-ray diffraction analysis, which are described elsewhere [17].

Introduction of molybdenum ions into the lattice of vanadium oxide demonstrating layered structure results in the transition of corresponding number of V(V) ions to V(IV) state. The change in the valence state of vanadium ions in the cationic sublattice results in significant decrease of the vibration energy of the $\text{V}=\text{O}$ group [20], this shift being known to be an indicative of the solid solution formation [17]: thus, fig. 1 evidenced that in the case of $0.6\text{MoO}_3 : 0.4\text{V}_2\text{O}_5$ this band is observed at 975 cm^{-1} (cf. 1009 cm^{-1} for the same vibrations in the case of bare V_2O_5).

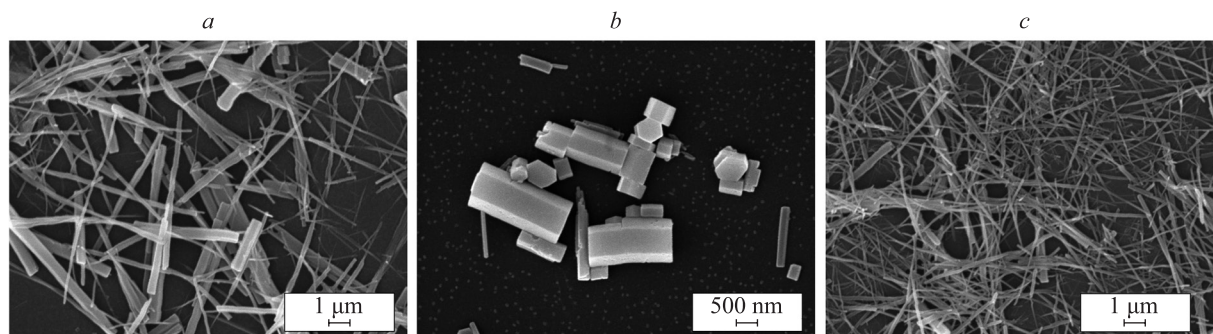


Fig. 2. SEM images of dispersions of $x\text{MoO}_3 : (1-x)\text{V}_2\text{O}_5$ oxide particles ($x = 0.25$ (a); $x = 0.4$ (b); $x = 0.75$ (c))

Due to the paramagnetic properties inherent in V(IV) centres, it is possible to trace the dynamic of mixed-oxide autoreduction employing EPR spectroscopy. The comparison of the integrated spectra with standard sample with known number of spins (see table) evidenced that the concentration of V(IV) centres in the $\text{MoO}_3 : \text{V}_2\text{O}_5$ mixed oxides exhibits an increase with MoO_3 loading. Number of V(IV) centres reaches its maximum at $1-x \approx 0.6$ and then tends to decline as the result of decrease of the relative content of V_2O_5 in the mixed oxide.

Number of spins in $\text{MoO}_3 : \text{V}_2\text{O}_5$ mixed oxides of different composition

$1-x$	$[\text{V(IV)}] \cdot 10^{-20}$, spin/g
1.0	0.78
0.9	1.42
0.75	3.2
0.5	3.6
0.25	2.8
0.1	1.0

The autoreduction, however, affects only a part of vanadium ions in the solid solution, whereas the major portion of vanadium ions remain in the diamagnetic V(V) state [21; 22]. On the other hand, the paramagnetic V(IV) centres exhibit a strong tendency towards clusterisation forming conductive domains in the $\text{MoO}_3 : \text{V}_2\text{O}_5$ particles [23]. It should be also noted that the charge exchange between V(IV)/V(V) and Mo(V)/Mo(VI) redox centres in the molybdenum – vanadium oxide solid solutions leads to complete oxidation of Mo(V) centres (these Mo(V) centres typically arises during the polycondensation synthesis). Therefore, in $\text{MoO}_3 : \text{V}_2\text{O}_5$ solid solution only V(IV) centres (both individual ones and those bound into the clusters) appear to be responsible for catalytic activity of $\text{MoO}_3 : \text{V}_2\text{O}_5$ particles towards nickel deposition.

Due to high concentration of V(IV) centres, the $\text{MoO}_3 : \text{V}_2\text{O}_5$ solid solution can behave as an effective redox catalyst facilitating the crystallisation of metal phase. Thus, the rate of nickel reduction under cathodic polarisation shows about 3-fold increase when passing from bare plating bath to the suspension plating bath containing dispersed $\text{MoO}_3 : \text{V}_2\text{O}_5$ particles. The redox catalysis results in the multi-centre nucleation of metal phase at the surface of $\text{MoO}_3 : \text{V}_2\text{O}_5$ particles that evidenced by SEM images in fig. 3. Such nucleation mode facilitates the overgrowing of the embedded particles with galvanic deposit and leads to the enhanced number of nickel nuclei capable of growing. The electron microscopic analysis reveals that the medium size of nickel grains grown at the surface of $\text{MoO}_3 : \text{V}_2\text{O}_5$ particles appears to be 2–3 times smaller as compared to the medium size of crystallites grown directly at the nickel surface (see fig. 3).

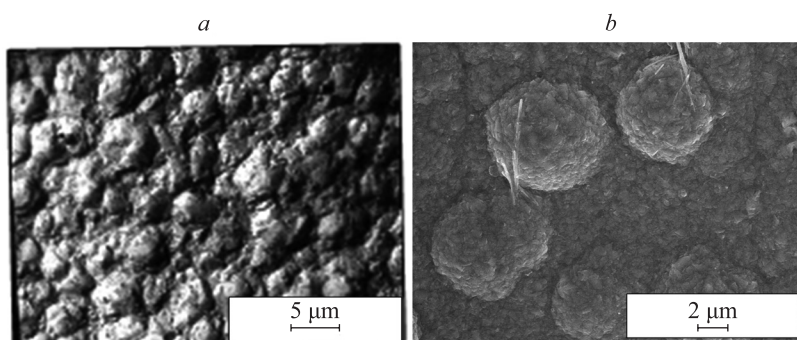


Fig. 3. SEM images of the surface of nickel (a) and $\text{Ni} - 0.6\text{MoO}_3 : 0.4\text{V}_2\text{O}_5$ (b)

The AFM images of Ni – MoO₃ : V₂O₅ composite provide an evidence that the effective nucleation and growth of metal grains at MoO₃ : V₂O₅ particles with high concentration of redox centres considerably affects the morphology of the resultant nickel matrix (fig. 4).

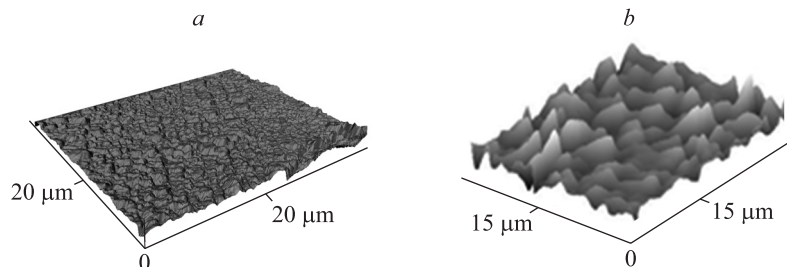


Fig. 4. AFM images of the surface of nickel (a) and Ni – 0.6MoO₃ : 0.4V₂O₅ (b)

It is seen from fig. 5 that even at low degree of substitution the size of nickel crystallites in the galvanic deposit diminishes and the well-developed surface relief is formed that manifests itself in higher fractal dimension of the resultant surface as compared to the surface of bare nickel deposited under similar conditions (the fractal dimensions thus enable very sensitive statistical index of complexity in surface relief). On the other hand, at high MoO₃ loading ($x = 0.75$) fine crystallites forming the galvanic deposit tend to agglomerate yielding micron-sized aggregates that results in some decrease of fractal dimension of the surface of metal-matrix composite surface (see fig. 5).

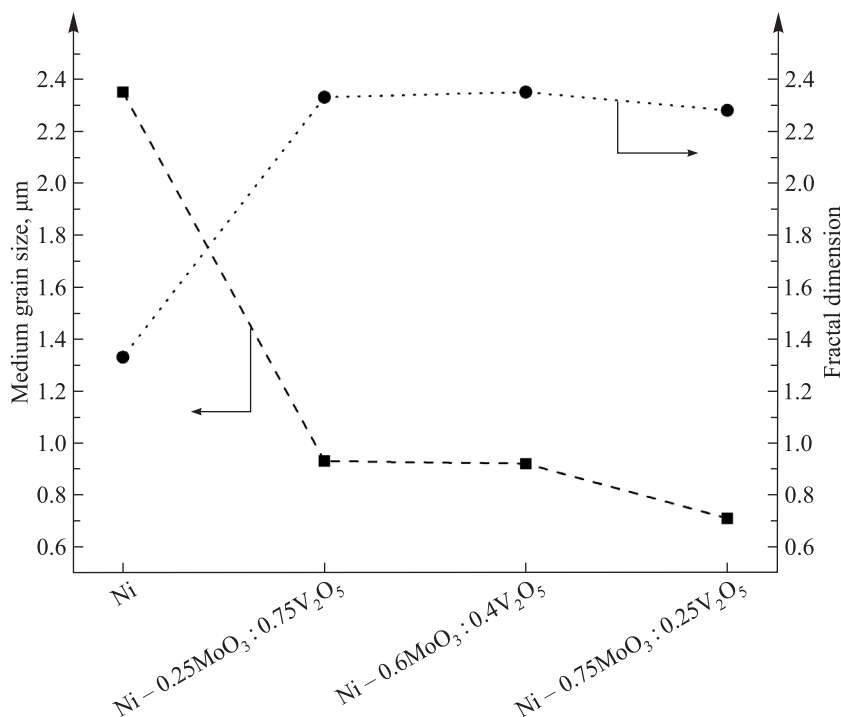


Fig. 5. Dependence of the medium size of nickel grains and fractal dimension of a surface for galvanic nickel and Ni – x MoO₃ : (1 – x)V₂O₅ composite films of different composition

The experimental results described above suggest that by choosing the composition of incorporated molybdenum – vanadium mixed oxide used for composite electrodeposition it is possible to turn the concentration of redox centres in MoO₃ : V₂O₅ oxide phase as well the degree of clusterisation of V(IV) centres. This permits one to exert an effective control over the growing of nickel deposit thus affecting the compactness of metal matrix and the size of metal crystallites forming the composite film. The latter factor has a pronounced effect both on mechanical properties of metal-matrix composite and on its corrosion resistance. Thus, it is seen from fig. 6 that the wear resistance exhibits considerable (about 3-fold) increase when coming from bare galvanic nickel to Ni – 0.6MoO₃ : 0.4V₂O₅ composite coating. However, at lower and higher MoO₃ loadings MoO₃ : V₂O₅ mixed oxide particles yield composites demonstrating poor wear resistance that can be attributed to the effects of dispersion wearing.

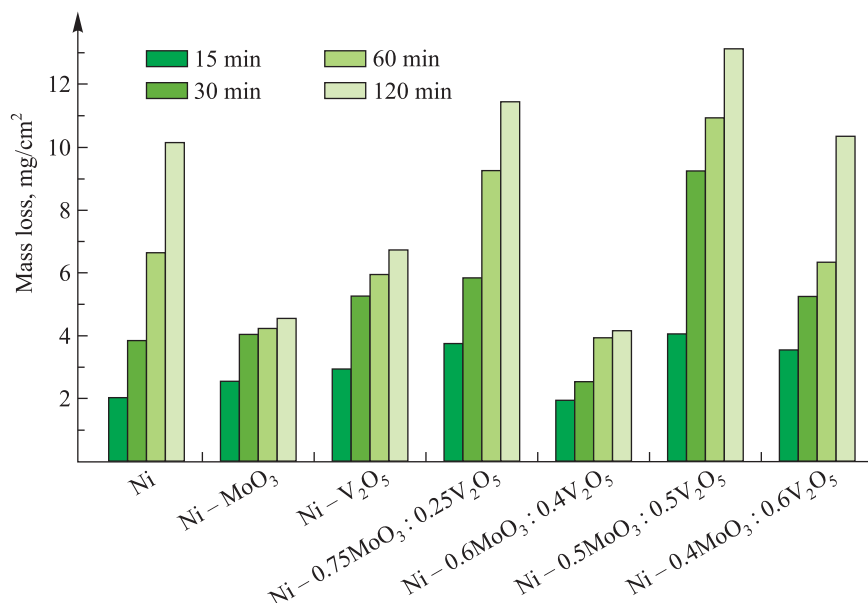


Fig. 6. Wear resistance of galvanic nickel and nickel-matrix composites of different composition

The compact and fine-crystalline nickel matrix in the case of Ni – MoO₃ : V₂O₅ composite ensures enhanced corrosion stability as evidenced by Nyquist plots shown in fig. 7: the Ni – 0.6MoO₃ : 0.4V₂O₅ coating demonstrates higher impedance as compared to bare nickel evidencing that the inclusion of MoO₃ : V₂O₅ particles with enhanced redox activity reduces its corrosion susceptibility.

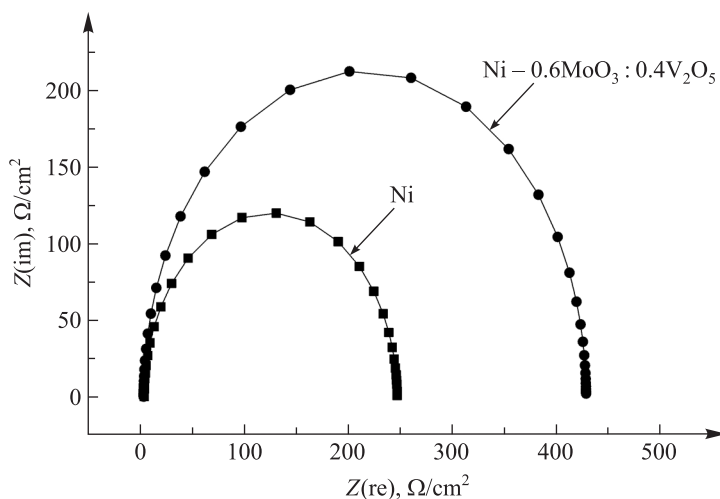


Fig. 7. Electrochemical impedance spectra of bare galvanic nickel and Ni – 0.6MoO₃ : 0.4V₂O₅ composite in 0.5 mol/L NaCl aqueous solution used as the corrosion medium. Measurements were done under open circuit potentials

Thus, by tailoring the redox activity of molybdenum – vanadium oxide particles (through the variation the Mo : V mole ratio) it is possible to exert an effective control over the electrochemical nucleation and growth of metal phase that yields metal-matrix composites with the enhanced wear and corrosion stability.

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Received 02.02.2026 / accepted 13.02.2026.