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ELECTROMAGNETIC-SHIELDING EFFECTIVENESS AND FRACTURE BEHAVIOR OF LAMINATED ($Ni–NiAl_3$) COMPOSITES

UČINKOVITOST ELEKTROMAGNETNE ZAŠČITE IN OBNAŠANJE PRI LOMU LAMINIRANEGA KOMPOZITA ($Ni-NiAl_3$)

Tuba Yener[1], Suayb Cagri Yener[2], Sakin Zeytın[1]

[1]Sakarya University, Engineering Faculty, Department of Metallurgy and Materials Engineering, Serdivan, Sakarya, Turkey
[2]Sakarya University, Engineering Faculty, Department of Electrical and Electronic Engineering, Serdivan, Sakarya, Turkey
syener@sakarya.edu.tr

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In this research $Ni–NiAl_3$ multilayer composites were produced through reactive sintering in an open atmosphere using Ni and Al foils with a 250-μm initial thickness. The sintering was performed at 700 °C under 2 MPa of pressure for 6 h. The microstructure and phase characterizations of the samples were performed. The hardness values of samples were determined using the Vickers indentation technique for the intermetallic and metallic regions as 765±60 HV and 90±10 HV, respectively. For the mechanical examinations, a perpendicular load was applied to the composite in order to observe the fracture behavior of the metallic-intermetallic laminate composites. SEM fracture surface analyses indicated that cracks initiated in the intermetallic region, and the crack propagation stopped when it reaches the ductile nickel phase. In addition, shielding-effectiveness measurements were performed. The MIL composite exhibits over 50 dB electromagnetic-shielding effectiveness against a very wide frequency range, from a few GHz to over 18 GHz.

Keywords: intermetallics, MIL composites, fracture behavior, electromagnetic interference shielding

V raziskavi so bili izdelani $Ni–NiAl_3$ večplastni kompoziti z reakcijskim sintranjem na atmosferi in z uporabo Ni- in Al-folij z začetno debelino 250 µm. Sintranje je bilo 6 h na 700 °C, pri tlaku 2 MPa. Na vzorcih je bila izvedena karakterizacija mikrostrukture in faz. Trdota vzorcev je bila določena po Vickersu, 765±60 HV, za področja intermetalnih faz in 90±10 HV pri osnovi. Za mehanske preiskave je bila uporabljena navpična obremenitev, za opazovanje obnašanja kompozita pri lomljenju kovinskih in intermetalnih lamel. SEM-preiskave prelomov so pokazale, da je začetek razpoke v področju intermetalne faze in da se širjenje razpoke ustavi, ko pride v duktilno fazo niklja. Izvedene so bile tudi meritve učinkovitosti zaščite sevanja. MIL kompozit kaže učinkovitost pred elektromagnetnim sevanjem, višjo od 50 dB v zelo širokem območju frekvenc od nekaj GHz do preko 18 GHz.

Ključne besede: intermetalne zlitine, MIL kompoziti, obnašanje pri lomu, elektromagnetna interferenčna zaščita

1 INTRODUCTION

Layered metallic-intermetallic laminate (MIL) composites are a new multifunctional materials group based on open air reactive sintering of chemically active metal foils under pressure.[1,2] Laminate composites are being intensively studied for a number of potential applications: electronic devices, structural components, armor, etc. Ceramic–ceramic, metal–ceramic, metal–metal, metal–ceramic–intermetallic and metal–intermetallic systems have shown desirable properties.[3–5] They are designed to optimize the desirable mechanical properties of intermetallics by incorporating layers of ductile reinforcement.[6] The combination of these types of materials makes the MIL composites candidates for the armament industry as armor materials that require improved mechanical and electromagnetic properties.[1,6,7]

In particular, nickel–tri-nickel aluminide ($Ni–NiAl_3$) metal–intermetallic laminate (MIL) composite systems have a great potential for aerospace, automotive and military applications because of their combination of high strength, toughness and stiffness at a lower density than monolithic titanium or other laminate systems.[7,8] Intermetallics of NiAl and $NiAl_3$ have a high melting point, a low density, high strength, good corrosion and oxidation resistance at high temperature.[9,10] The nickel-aluminum system is one of the most well known in terms of the formation of intermetallic phases. This system is also a priority among laminate composite systems.[2,5,11]

The aim of the present study is to synthesize nickel-nickel aluminide metallic-intermetallic composites and analyze their mechanical, fracture and electromagnetic shielding behaviors. The organization of the paper is as follows. After this introduction, in the second section the methodology and production of materials ear summarized. In the third section the experimental results are presented. In this section, the fracture behavior in terms of "physical-shielding" and then electromagnetic-shielding behavior of the composites are provided after experimental processes. Finally, the paper ends with a conclusion section.

2 METHODOLOGY AND EXPERIMENTAL PART

2.1 Materials and method

The MIL process consists of stacking commercial-purity Ni and Al foils in alternating layers. The properties of the foils are listed in **Table 1**.

Table 1: Properties of foils used in experiments
Tabela 1: Lastnosti folij, uporabljenih pri preizkusih

Foil	Ni	Al
Thickness (μm)	250	250
Purity (%)	99.5	99.5
Stack number	6	5

The nickel- and aluminum-foil dimensions were initially selected to completely consume the aluminum in forming the intermetallic compound with alternating layers of partially unreacted Ni metal. Each foil sheet was prepared as 10 mm × 10 mm and 60 mm × 40 mm rectangular pieces for mechanical and electromagnetic experiments, respectively. Contamination on the surface of the foils was cleaned using ethanol. After drying rapidly, they were laminated alternatively into nickel/aluminum multilayer samples. Each stack consisted of 6 nickel and 5 aluminum foils, as indicated in **Table 1**. An initial pressure of 2 MPa is applied at room temperature to ensure good contact between the foils. A schematic representation of the Ni-Al stacks is shown in **Figure 2a**. The sintering process was applied in the open air, in an electrical resistance furnace at 700 °C for 6 h. After sintering, samples were ground and polished using standard metallographic techniques.

2.2 Characterization

Microstructure analyses of the composites were performed with a JEOL JSM-5600 model scanning electron microscope (SEM). The presence of phases formed in the sintered samples was determined by energy-dispersive spectroscopy (EDS). The microhardness of composites was determined using a Leica WMHT-Mod model Vickers hardness instrument under an applied load of 300 g for the intermetallic zone, and 100 g for the metallic zone. The composition of the phases was determined by comparing the results of the microprobe analysis with the data in the binary Ni–Al phase diagram (**Figure 1**).[12]

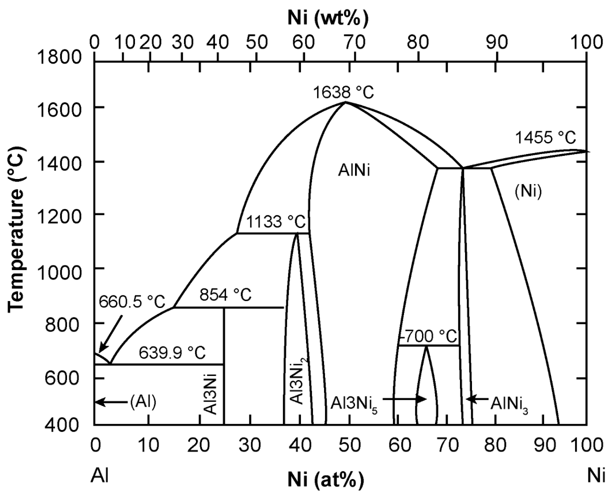

Figure 1: The Ni–Al binary phase diagram[12]
Slika 1: Binarni fazni diagram Ni-Al[12]

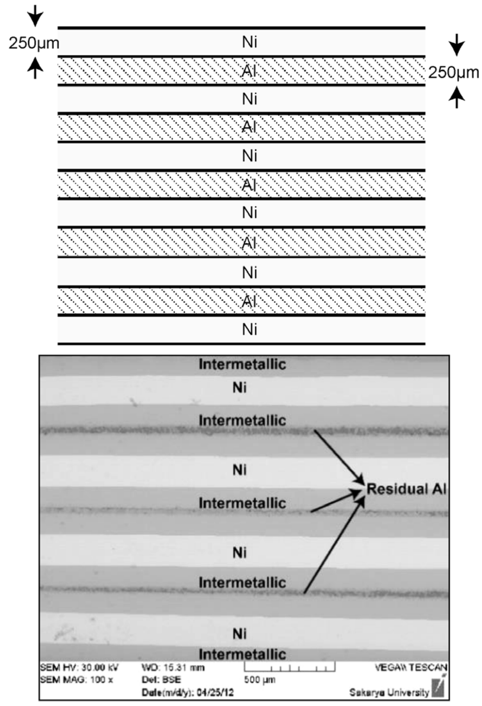

Figure 2: a) Nickel-aluminum foils stack, b) SEM micrograph of laminated composites produced at 700 °C/6h
Slika 2: a) Sestav nikelj-aluminijevih folij, b) SEM-posnetek laminiranega kompozita, izdelanega pri 700 °C/6 h

3 EXPERIMENTAL PROCESSES AND RESULTS

3.1 SEM-EDS Analysis

Figure 2b presents the cross-sectional micrographs of representative laminated composites. The presence of

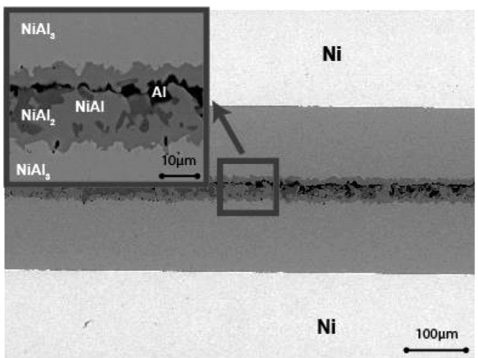

Figure 3: SEM-EDS analyses of Ni-$NiAl_3$ composites sintered at 700 °C/6h
Slika 3: SEM-EDS analize Ni-$NiAl_3$ kompozita po sintranju 6 h na 700 °C

different regions indicates the different phases in the composites. It can be seen that the laminated composites consist of unreacted Ni layers (gray regions) and the formed intermetallic $NiAl_3$ layers (dark regions). Moreover, the laminated composites are well-bonded and remain nearly fully dense. The nickel aluminide phase occurs due to the thermodynamics of the reaction between Ni and Al. The existence of liquid Al phase plays important roles in the nucleation and growth of NiAl particles and the eventual formation of continuous alternative intermetallic layers.

3.2 Mechanical fracture behavior and hardness

Intermetallics and ceramics, in general, have very little or no dislocation motion, and, hence, exhibit very little inherent or intrinsic crack-propagation resistance.[3] By using laminate design and proper composites, it is aimed to produce intermetallic $NiAl_3$ phase during the process to give a high hardness to the composite, while unreacted nickel provided moderate ductility.

Due to the deflection of cracks along the Ni/NiAl interfaces, a non-catastrophic fracture was observed in the laminated composites. A weak delamination and debonding is seen at the metallic nickel and the intermetallic layers interface. In a large number of cleavage cracks present in the brittle intermetallic layer. Despite the severe plastic deformation, the nickel layer was not torn. This clearly demonstrates the effect of crack stopping of the ductile reinforcing phases (**Figure 4**).

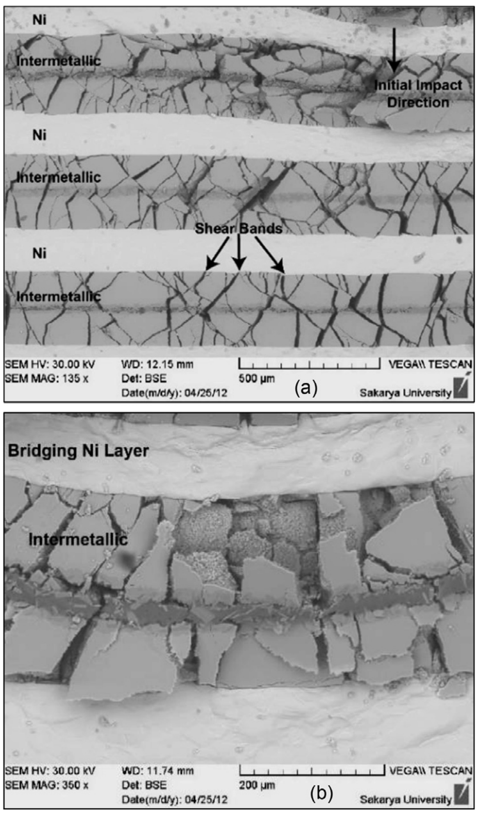

Figure 4: Cross-sectional micrographs of Ni-$NiAl_3$ composites after impact effect: a) 135× , b) 350×

Slika 4: Posnetek preseka kompozita Ni-$NiAl_3$ po udarcu: a) 135×, b) 350×

When it comes to hardness, the values of samples were determined by using the Vickers indentation technique for intermetallic and metallic region as 765±60 HV, 90±10 HV, respectively, whereas the hardness of metallic aluminum and nickel, respectively, is about 45 HV and 90 HV

3.3 Electromagnetic-shielding effectiveness

Electromagnetic interference can lead to adverse consequences, such as malfunction or crashing of electronic systems and computers, unintentionally firing of electrically explosive devices, or be the cause of the loss of secret information to an enemy. In this respect, it is essential to protect devices from disruptive electromagnetic signals to guarantee their functionality in stable operating conditions. It is also obvious that the electromagnetic shielding is vital in military applications.[13–15]

Shielding effectiveness is the ratio of impinging energy to the residual energy. When an electromagnetic wave passes through a shield, absorption and reflection take place. The residual energy is part of the remaining energy that is neither reflected nor absorbed by the shield, but emerges from the shield. Shielding effectiveness (*SE*) is the ratio of the field before and after

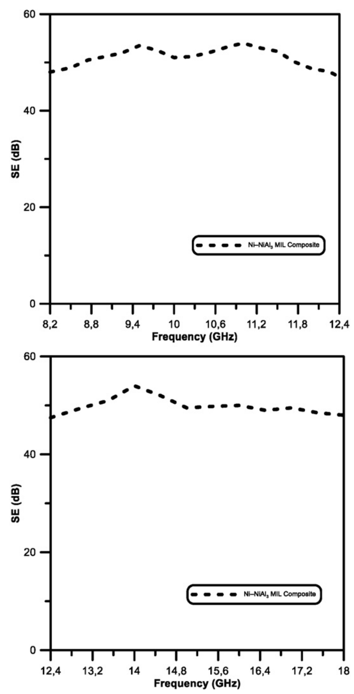

Figure 5: Electromagnetic-shielding effectiveness characteristics of laminated Ni–$NiAl_3$ composites a) X Band (8.2–12.4 GHz), b) Ku Band (12.4–18 GHz)

Slika 5: Značilnost učinkovitosti elektromagnetne zaščite laminiranega Ni-$NiAl_3$ kompozita a) X-pas (8,2 GHz-12,4 GHz), b) Ku-pas (12,4 GHz – 18 GHz) attenuation of the electric and magnetic fields and can be expressed as Equation (1):[15,16]

$$SE[dB] = 20\lg\left|\frac{E_i}{E_t}\right| \quad (1)$$

Where E_i and E_t refer to the transmitted and incident waves, respectively. Shielding effectiveness is a function of frequency, and from the Equation (1) it is measured in dB.

The shielding-effectiveness characteristics of laminated Ni–$NiAl_3$ composites have been measured and the results obtained are shown in **Figures 5a** and **5b** for the X band and Ku band, respectively.

From the results, the produced MIL composites exhibit around or more 50 dB of electromagnetic-shielding effectiveness against a very wide frequency range from 8.2 GHz to over 18 GHz. That shielding level means even 99.999 % of the incident power is prevented by the produced composites. These shielding-effectiveness levels indicate that laminated composites can be remarkable candidates for shielding application also thanks to their improved mechanical properties.

4 CONCLUSIONS

The conclusions of this research can be summarized as follows:

- By controlling the duration of the reactive-foil sintering process, composites can be fabricated in which a tailored amount of residual aluminum remains at the intermetallic centerline.
- Ni–$NiAl_3$ metal–intermetallic laminate (MIL) composites have been successfully synthesized by reactive-foil sintering technique in open air at 700 °C for 6 h under 2 MPa pressure. The laminated structure is well-bonded, nearly fully dense.
- Microstructural characterization by SEM and EDS indicates that NiAl, $NiAl_3$, Ni_2Al_3 are intermetallic phases in the composite.
- The hardness of the fabricated laminated composite was dramatically changed. Whereas the hardness of metallic aluminum and nickel, respectively, is about 45 HV and 90 HV, the hardness of intermetallic zone is approximately 765±60 HV.
- In this study the shielding effectiveness of laminated Ni–$NiAl_3$ composites was examined in a two-frequency band at GHz levels and the results obtained are shown. Around 50-dB shielding-effectiveness levels were reached experimentally from the measurements.
- Thus, experimental results obtained are promising for MIL composites to be appropriate candidate materials for military applications with their electromagnetic as well as mechanical properties.

5 REFERENCES

[1] K. S. Vecchio, Synthetic multifunctional metallic-intermetallic laminate composites, JOM, 57 (**2005**) 3, 25–31, doi:10.1007/s11837-005-0229-4

[2] B. Besen, M. Kalayci, T. Yener, S. Zeytin, Some Properties Of Ni-AlNi Metallic-Intermetallic Laminate Material, Journal of International Scientific Publications: Materials, Methods & Technologies, 7 (**2013**) 2, 390–396

[3] R. R. Adharapurapu, K. S. Vecchio, F. Jiang, A. Rohatgi, Fracture of Ti-Al3Ti metal-intermetallic laminate composites: Effects of lamination on resistance-curve behavior, Metallurgical and Materials Transactions A, 36 (**2005**) 11, 3217–3236, doi:10.1007/s11661-005-0092-5

[4] L. Peng, H. Li, J. Wang, Processing and mechanical behavior of laminated titanium–titanium tri-aluminide (Ti–Al_3Ti) composites, Materials Science and Engineering A, 406 (**2005**) 1, 309–318, doi:10.1016/j.msea.2005.06.067

[5] X. Yang, X. Peng, F. Wang, Size effect of Al particles on the structure and oxidation of Ni/Ni3Al composites transformed from electrodeposited Ni–Al films, Scripta Materialia, 56 (**2007**) 6, 509–512, doi:10.1016/j.scriptamat.2006.11.016

[6] A. Rohatgi, D. J. Harach, K. S. Vecchio, K. P. Harvey, Resistance-curve and fracture behavior of Ti–Al_3Ti metallic–intermetallic laminate (MIL) composites, Acta Materialia, 51 (**2003**) 10, 2933–2957, doi:10.1016/S1359-6454(03)00108-3

[7] Y. Cao, C. Guo, S. Zhu, N. Wei, R. A. Javed, F. Jiang, Fracture behavior of Ti/Al3Ti metal-intermetallic laminate (MIL) composite under dynamic loading, Materials Science and Engineering A, 637 (**2015**), 235–242, doi:10.1016/j.msea.2015.04.025

[8] K. H. Zuo, D. L. Jiang, Q. L. Lin, Mechanical properties of Al2O3/Ni laminated composites, Materials Letters, 60 (**2006**) 9–10, 1265–1268, doi:10.1016/j.matlet.2005.11.010

[9] L. Z. Zhang, D. N. Wang, B. Y. Wang, R. S. Yu, L. Wei, Identification of lattice vacancies in the B2-phase region of Ni–Al system by positron annihilation, Journal of Alloys and Compounds, 457 (**2008**) 1–2, 47–50, doi:10.1016/j.jallcom.2007.03.065

[10] F. L. Zhang, Z. F. Yang, Y. M. Zhou, C. Y. Wang, H. P. Huang, Fabrication of grinding tool material by the SHS of Ni–Al/diamond/dilute, International Journal of Refractory Metals and Hard Materials, 29 (**2011**) 3, 344–350, doi:10.1016/j.ijrmhm.2010.12.013

[11] C. T. Wei, V. F. Nesterenko, T. P. Weihs, B. A. Remington, H. S. Park, M. A. Meyers, Response of Ni/Al laminates to laser-driven compression, Acta Materialia, 60 (**2012**) 9, 3929–3942, doi:10.1016/j.actamat.2012.03.028

[12] ASM Handbook, Vol. 3: Alloy Phase Diagrams, ASM International, 2001

[13] P. Saini, M. Aror, Microwave Absorption and EMI Shielding Behavior of Nanocomposites Based on Intrinsically Conducting Polymers, Graphene and Carbon Nanotubes, Chapter 3, In: A. De Souza Gomes (Ed.), New Polymers for Special Applications, InTech, 2012, doi:10.5772/48779

[14] C. R. Paul, Introduction to electromagnetic compatibility, 2nd Edition, John Wiley & Sons, 2006, 184

[15] S. Geetha, K. K. Satheesh Kumar, C. R. K. Rao, M. Vijayan, D. C. Trivedi, EMI shielding: Methods and materials-A review, Journal of Applied Polymer Science, 112 (**2009**) 4, 2073–2086, doi:10.1002/app.29812

[16] O. Cerezci, S. Şeker, Ş. Yener, B. Kanberoğlu, M. H. Nişancı, Ev, Ofislerde GSM Frekanslı Radyasyondan Bireysel Korunma, EMANET, Yıldız Teknik Üniversitesi, Beşiktaş, İstanbul 2013, 372–376