

Fatigue shear testing of Nimonic 80A valve rods with metal-ceramic Ni-Al-Al₂O₃ composite heads

Andrej Opálek^{1*}, Karol Išdinský¹, Stanislav Kúdela Jr.¹, Pavol Štefánik¹,
František Simančík¹

¹*Institute of Materials and Machine Mechanics, Slovak Academy of Sciences, Bratislava, Slovak Republic*

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Abstract

This study focuses on the development and fatigue testing of a Ni-Al-Al₂O₃ metal-ceramic composite intended for application in the exhaust valve heads of internal combustion engines. The material was prepared by gas-pressure infiltration of molten aluminum into a porous skeleton (Ni, Al₂O₃, Al), which was pre-stabilized by oxidation annealing at temperatures up to 800 °C. A key aspect of the research was the evaluation of the shear strength and interface integrity between the developed composite and a Nimonic 80A nickel superalloy stem. Fatigue tests were conducted at temperatures ranging from room temperature up to 800 °C at a frequency of 20 Hz. The results demonstrated that the composite and its bond with the stem exhibit excellent stability; a specimen loaded with a maximum force of 2000 N at 800 °C withstood 10 million cycles without any observed mechanical damage. Microstructural analysis confirmed that cyclic thermal loading leads to the transformation of NiAl₃ and Ni₂Al₃ phases into more stable NiAl and Ni₃Al intermetallic phases. The developed material represents a progressive lightweighting alternative to conventional steel valves, capable of long-term resistance to extreme conditions in the automotive industry.

Key words: lightweighting materials, automotive industry, gas pressure infiltration, nickel aluminides, ceramic particles

1. Introduction

Lightweighting of components is one of the most crucial trends and innovative approaches in the modern automotive industry. It is not just about reducing the overall vehicle weight, but a comprehensive strategy that offers several fundamental advantages [1]. The most significant and economically important benefit is the reduction in fuel (or energy) consumption. Every saved kilogram directly translates into higher efficiency and lower emissions [2]. Another significant benefit is the reduced wear and tear of the vehicle and its components. Lower mass means smaller inertial forces, leading to less strain on brakes, tires, and chassis components, improved handling and maneuverability, and an extended vehicle's overall lifespan [3].

Lightweighting is especially vital for components within the vehicle's powertrain, which are subjected to extreme stress, high temperatures, and wear. Typical examples include intake and exhaust valves. These components must meet strict and often conflicting requirements such as high strength and stiffness, to withstand dynamic forces and pressures within the cylinder, wear resistance, to minimize wear during contact with other parts of the mechanism, good thermal conductivity (heat dissipation), for effective cooling and prevention of overheating, which could lead to material degradation and excellent resistance to chemical corrosion, for protection against the aggressive environment of combustion gases [4]. Increasing the thermal efficiency of modern internal combustion engines demands materials capable of withstanding extreme mechanical and corrosive environments at tempera-

*Corresponding author: e-mail address: andrej.opalek@savba.sk



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tures exceeding 800°C, which severely limits the use of standard automotive steels [5]. Intermetallic compounds based on the Ni-Al system, particularly NiAl and Ni₃Al, have emerged as progressive candidates due to their high melting points, excellent oxidation resistance, and low density compared to conventional superalloys [6].

The conventional materials used for intake and exhaust valves in internal combustion engines are primarily specialized steel alloys [7, 8]. However, their compositions differ significantly to accommodate each valve's operating conditions. Intake valves operate at considerably lower temperatures (200 to 550°C) because the incoming, cooler air-fuel mixture cools them. The key requirements are high strength, good anti-friction properties, and durability. Common conventional materials include martensitic steels, iron-chromium alloys that can be hardened by heat treatment (quenching and tempering). They offer good strength and wear resistance. Then, carbon steel alloys are used in older or standard engines for basic applications. High-performance stainless-steel alloys, used in more stressed applications, are sometimes selected for the same alloy as the exhaust valve for uniformity. Exhaust valves are subjected to much more severe conditions, constantly blasted by hot combustion gases and typically operating at 650 to 800°C. They require excellent heat resistance, corrosion resistance, and high hot hardness. Common conventional materials include austenitic stainless steels, chromium-nickel-silicon alloy steels, and bimetallic valves. The first one is the most popular traditional choice for performance and heavy-duty engines. They contain higher percentages of chromium (for corrosion resistance) and nickel (for high-temperature strength). They cannot be hardened by quenching but maintain strength at high heat. The second one is specifically formulated for high-temperature and high-stress conditions. The third one is a standard modern design in which the valve head (exposed to extreme heat) is made of heat-resistant austenitic steel, and the valve stem (exposed to wear) is made of high-strength, wear-resistant martensitic steel. The two sections are joined, often by friction welding. For extreme applications (turbocharged, racing, or heavy-duty), superalloys are frequently used, such as nickel-based superalloys (e.g., Inconel or Nimonic). These offer superior strength and thermal resistance at very high temperatures. Titanium alloys are prized for their high strength-to-weight ratio and low density, making them ideal for high-RPM racing engines where reducing mass is critical, despite the higher cost. Valve faces are often reinforced with hard materials, such as Stellite (a cobalt-chromium alloy), to improve wear resistance at the valve seat, and stems may be chromium-plated or nitrided to enhance surface hardness and reduce friction.

However, even with all the demanding criteria for

intake or exhaust valves, innovative and advanced materials can be used. One of the primary candidates for high-performance components is nickel aluminides reinforced (strengthened) with Al₂O₃ (aluminum oxide) ceramic particles. These materials, known as Metal Matrix Composites (MMCs), combine the properties of a metal matrix (good toughness, thermal and electrical conductivity) with those of ceramic reinforcement (extreme hardness, strength, and resistance to heat and wear). Nickel aluminides are a group of intermetallic compounds that are crucial in the aerospace and automotive industries, especially where extreme high-temperature resistance is required. Among the key properties and advantages of nickel aluminides are excellent strength at high temperatures. Unlike many conventional alloys, Ni₃Al retains high strength and stiffness even at temperatures exceeding 800°C. This is critical for exhaust valves directly exposed to hot combustion gases. When exposed to heat, they form a stable, protective Al₂O₃ layer on the surface, preventing further degradation and chemical corrosion. Compared to traditional nickel-based alloys or steels, intermetallic aluminides have a lower density, which directly supports the goal of component lightweighting. Their structure is stable, ensuring the long-term lifespan of components without changes in mechanical properties. Nickel aluminide alone is excellent at high temperatures, but to maximize resistance to wear and tear and increase hardness, Al₂O₃ ceramic particles are embedded within it. Rigid ceramic particles dispersed in the metal matrix significantly hinder dislocation movement and microcrack formation, thereby increasing the material's overall hardness and resistance to wear. Reinforcement also improves the strength and stiffness of the composite, which is essential for components under high cyclic loading. The result is the Ni₃Al/Al₂O₃ composite, which combines the best properties of metal (toughness, thermal conductivity) and ceramic (hardness, heat resistance).

In the context of valve manufacturing technologies, one of the most effective methods for producing composites with high ceramic phase content and minimal porosity, ensuring maximum performance, is pressure infiltration of molten metal [7–9]. First, an appropriately designed prefabricated nickel-ceramic metal skeleton (a preform) is created. This skeleton contains pores and channels that are subsequently filled with the molten metal. The molten metal alloy (in this case, nickel aluminide) is injected under high pressure into the ceramic skeleton's pores and interstitial spaces. The pressure ensures that the metal reaches even the most minor cavities, achieving perfect bonding between the ceramic particles and the metal matrix. The result is a lightweight, strong, and thermally stable composite material, ideal for extreme loads in automotive engines.

Table 1. Specification of powders used in the study

Powder	Purity (%)	Type and producer	Size particles (μm)	D_{50} (μm)
Nickel (Ni)	99.30	Metco 56C-NS, OC Oerlikon Corporation, Pfäffikon, Switzerland	< 75	69.91
Aluminum oxide (Al_2O_3)	95.50	Metco Amdry 6060, OC Oerlikon Corporation, Pfäffikon, Switzerland	< 45	33.17
Aluminum (Al)	95.50	Alpoco A1050, AMG Alpoco UK Limited, UK	< 63	30.32

In this work, we prepared and fatigue-tested a Ni-Al- Al_2O_3 -based metal-ceramic composite material suitable for the production of an internal combustion engine exhaust valve head. The shear strength of the interface between the prepared composite and Ni alloy was also tested.

2. Material and methods

2.1. Preparation of test specimens for fatigue loading of an intermetallic composite with a centrally located Ni alloy core

Test specimens were fabricated to evaluate specific fatigue properties of the developed material, as well as the shear strength of the bond between the composite material pressed onto a nickel superalloy (Nimonic 80A, manufactured by Special Metals Corporation, consisting of approximately 76 % Ni, 20 % Cr, 2.4 % Ti, and 1.4 % Al) stem. Nimonic 80A was selected because nickel superalloys are currently the standard material for producing exhaust valves. The Nimonic 80A stem featured a diameter of 6 mm and a length of 60 mm as detailed in the fixture schematics. During uniaxial pressing, the stem was precisely held inside a centralized guiding die assembly to ensure symmetrical compaction of the surrounding powder mixture around the core.

Initially, a mixture of nickel powder containing 12.87 wt.% Al_2O_3 (corresponding to 25 vol.%) was prepared. This composition was selected based on prior experiments showing that higher Al_2O_3 content led to reduced compact cohesion after pressing. The following powders were used for green body preparation:

To enhance compressibility, 3 g of aluminum powder was added to the Ni- Al_2O_3 blend, exploiting aluminum's relatively ductile nature. As a metallic plasticizer, aluminum significantly improves pressability compared to conventional organic binders, such as vegetable oils, citric acid, or starch. This amount was determined to be optimal for increasing green density without substantially reducing the required porosity. The final volumetric composition of the resulting blend (hereinafter referred to as the "powder mixture") was 68.76 % Ni, 22.95 % Al_2O_3 , and 8.29 % Al.

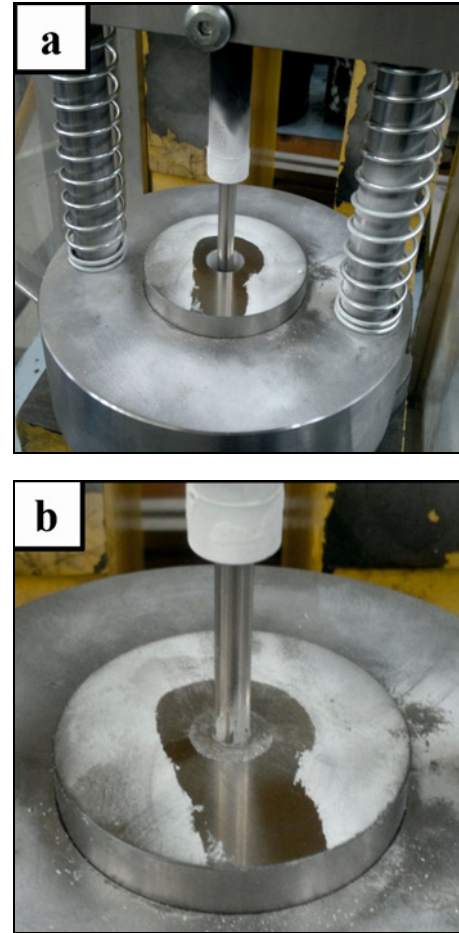


Fig. 1. Die for pressing the powder mixture onto the Ni-alloy stem. Overall view (a) and alignment of the powder around the stem (b).

The particle size distribution of each constituent powder was measured by laser diffraction using an Analysette 22 NanoTec (Fritsch, Germany) in water. The powders used, and their corresponding D_{50} values, are summarized in Table 1.

Finally, the powder mixture was homogenized for 30 minutes using a Turbula T2F mechanical mixer (WAB, Muttenz, Switzerland). The homogenized blend was then uniaxially pressed at a pressure of 250 MPa (see Figs. 1, 2a). The resulting green compacts were subsequently subjected to oxidation

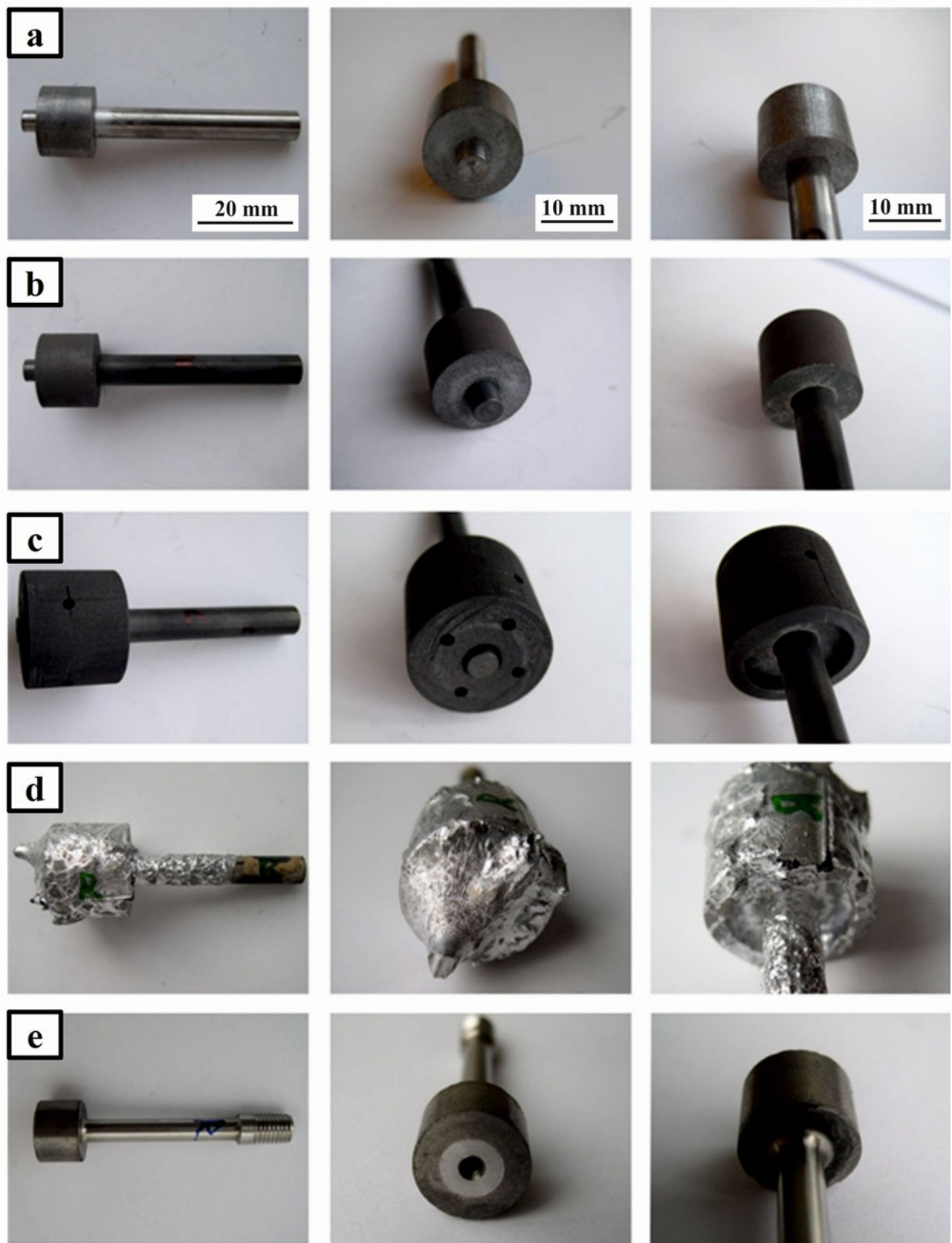


Fig. 2. Procedure for preparing a specimen for fatigue loading: pressed compact with a core (a), specimen after oxidation (b), placement of the specimen into the graphite mold (c), specimen together with the mold after infiltration (d), and view of the test specimen after grinding (e).

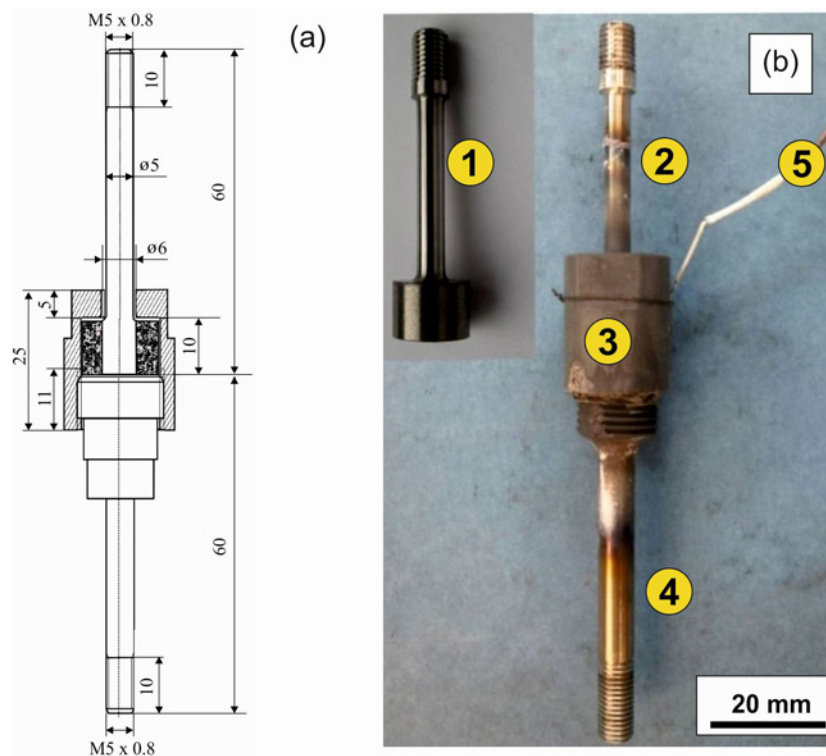


Fig. 3. Schematic of the specimen arrangement within the fixture (a) and real photo (b), a view of the test specimen (1) and (2) positioned in the upper (3) and lower (4) sections of the Ni-alloy fixture with an attached thermocouple (5).

annealing at temperatures up to 800 °C to suppress the exothermic reaction between Ni and Al particles, leading to the formation of a NiO barrier layer [10]. The annealed compacts were then placed into porous graphite crucibles with lateral perforations designed to facilitate the penetration of molten aluminum (Fig. 2c). Infiltration was carried out at 750 °C for 300 s under an argon atmosphere at 5 MPa using molten aluminum (Fig. 2d). After infiltration, the compacts were solidified and finally machined by precision turning and cylindrical grinding to obtain the specific geometries required for fatigue performance evaluation (Fig. 2e).

2.2. Process of fatigue tests of materials

Fatigue testing of the prepared composite and the interface to Ni alloy was performed using an MTS 312.21 servo-hydraulic testing system (100 kN, SPMGS077) equipped with an MTS 661.20F-01 load cell (25 kN, SPMGS121) and an MTS 653.02A high-temperature furnace capable of operation up to 1200 °C (SPMGS 131.5). A total of five test specimens were systematically analyzed under different regimes to establish trends. The loading frequency (20 Hz) and the minimum load (500 N) were kept constant across all tests, while the maximum load was adjusted to achieve fatigue lives of 2000–6000 cycles. Tests were conducted at temperatures from room temperature up

to 800 °C. The total number of applied cycles ranged from 1,000,000 to 10,000,000. A schematic of the specimen mounting and its placement within the test fixture is shown in Fig. 3.

2.3. Microstructural and chemical analysis

The microstructure was analyzed by SEM-EDS using a JEOL JSM 6610 scanning electron microscope (JEOL, Tokyo, Japan). *AZtec software* (Oxford Instruments, UK) was employed to evaluate the EDS peak intensities (in keV) of individual elements (Ni and Al). Based on the Ni-Al atomic percent ratios, the corresponding nickel aluminide intermetallic phases were identified.

3. Results and discussion

The effect of fatigue loading of the intermetallic composite in air at temperatures up to 800 °C on its microstructure, as well as on the quality of the bond between the composite and the Ni-alloy stem, was investigated.

3.1. Interfacial characterization prior to testing

To evaluate the bonding mechanisms that enabled

Table 2. Testing conditions and obtained results for individual specimens

Tested samples	Temperature (°C)	Min. force (N)	Max. force (N)	Number of cycles	Notes
F01-01	RT	500	2000	2000000	–
	RT	500	3000	2000000	–
	500	500	2000	2000000	–
	500	500	3000	1000000	–
	500	500	4000	1000000	–
	500	500	5000	360000	Nimonic-composite interface breaks off
F01-02	500	500	2000	1000000	–
	500	500	3000	1000000	–
	500	500	4000	165000	Break off at the upper thread of the Nimonic mount
F02-01	500	500	2000	1000000	–
	500	500	3000	1000000	–
	500	500	4000	1000000	–
	500	500	5000	1000000	–
	500	500	6000	430000	Break off at the upper thread of the Nimonic mount
	500	500	6000	430000	Break off at the upper thread of the Nimonic mount
F03-01	700	500	1000	1000000	–
	700	500	1500	1000000	–
	700	500	2000	1000000	–
	700	500	2500	1000000	–
	700	500	3000	1000000	–
	700	500	3500	1000000	–
	700	500	4000	800000	Stuck in the composite/test holder fixture
	700	500	4000	800000	Stuck in the composite/test holder fixture
F03-02	800	500	2000	10000000	OK

high fatigue performance, the initial interface between the central Nimonic 80A core, the transition layer, and the surrounding composite head was thoroughly characterized prior to fatigue cycling. As shown in the SEM micrograph in Fig. 7, a distinct, continuous reaction zone with an average thickness of approximately 150 μm was successfully formed during the gas-pressure infiltration process. This transition layer consists predominantly of newly formed Ni-aluminide phases, providing a strong metallurgical anchor between the superalloy stem and the composite. The reaction kinetics of similar systems, where a solid nickel core interacts with molten aluminum under varying exposure times and temperatures, drive the development of this interfacial zone and dictate the phase layer growth [11].

To map the elemental distribution across this boundary prior to loading, a comprehensive SEM-EDS chemical analysis of the diffusion profile was conducted. The resulting EDX mapping (Fig. 8) confirmed active multi-component interdiffusion of elements across the interface. Most notably, a distinct

migration of chromium (Cr) from the Nimonic 80A superalloy matrix directly into the newly formed interfacial reaction zone was observed, alongside the balanced interdiffusion of Ni and Al. These results are in good agreement with the multi-component interdiffusion mechanisms and layer growth rates previously systematically quantified by the authors [11].

The specimens were subjected to fatigue tests that differed in test temperature, the mode of maximum load increase, and the number of cycles applied under specific conditions (Table 2).

In the first test (specimen F01-01), the specimen was initially exposed to a maximum load of 2000 N at room temperature (RT) for 2 million cycles, followed by 3000 N for an additional 2 million cycles. Subsequently, the temperature was increased to 500 °C, and the maximum load was progressively increased in steps of 1000 N, from 2000 up to 5000 N. The number of cycles at each maximum load ranged from 1 million to 2 million. Failure of the Nimonic-composite interface occurred after 360,000 cycles at a maximum load of 5000 N (Fig. 4a, specimen F01-01 in Table 2). Detailed

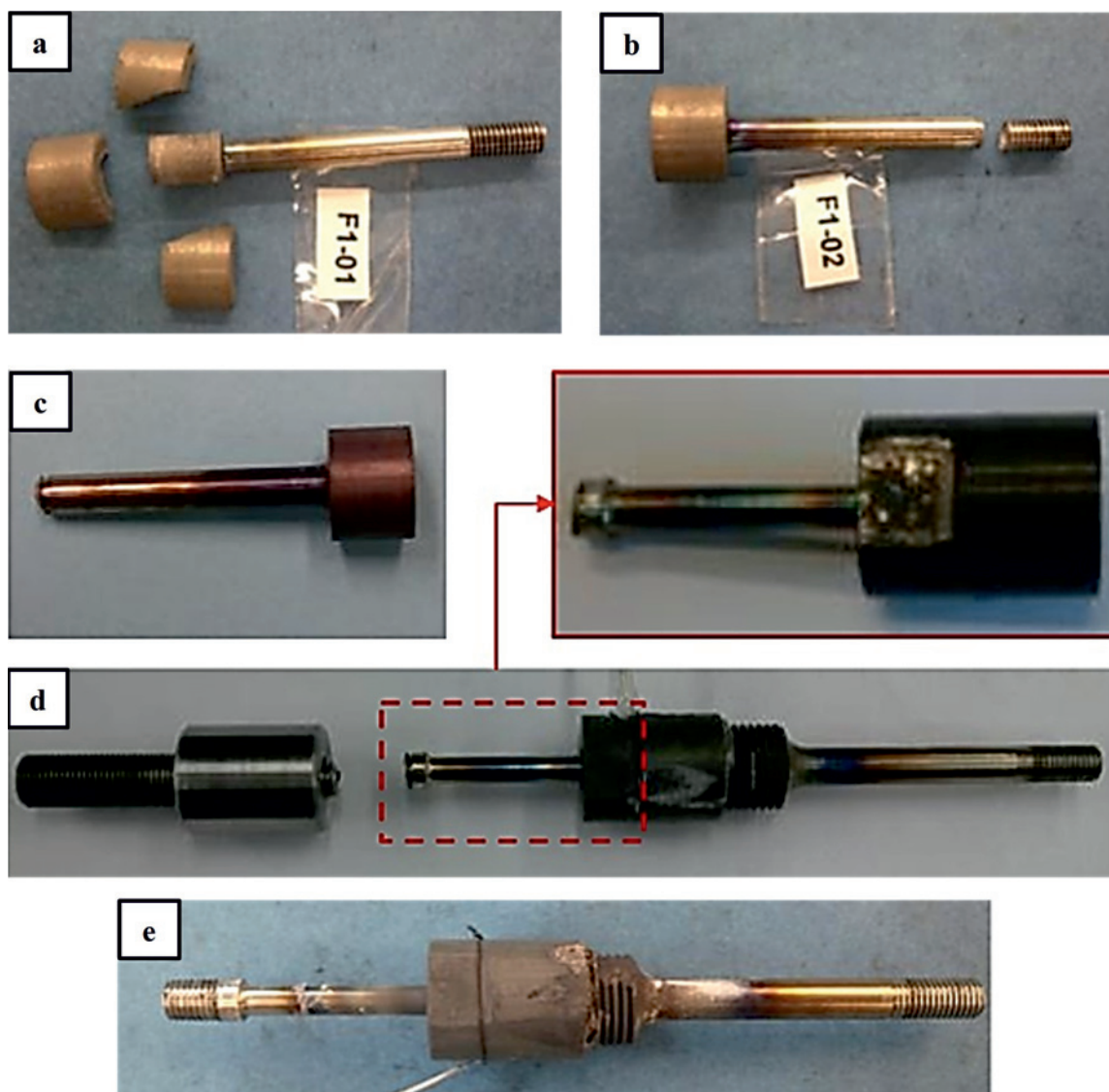


Fig. 4. Failure of the Nimonic-composite interface occurred after 360,000 (a), failure occurred at the upper threaded section of the Nimonic fixture after only 165,000 cycles (b), failure occurred at the upper threaded section of the Nimonic fixture after 430,000 cycles (c), seizure occurred at the composite – test fixture interface at a maximum after 800,000 cycles, no mechanical damage was observed even after 10 million cycles (e.)

microstructural analysis of this specimen revealed increased porosity within the composite volume, most likely resulting from insufficient Infiltration of the pre-form.

In the second test (specimen F01-02), the temperature was kept constant at 500 °C throughout the experiment. The maximum load was progressively increased by 1000 N from 2000 to 4000 N, with 1 million cycles applied at each load level. However, at a maximum load of 4000 N, failure occurred at the upper threaded section of the Nimonic fixture after only 165,000 cycles (Fig. 4b, specimen F01-02 in Table 2).

In the third test (specimen F02-01), the temperature was again maintained at 500 °C. The maximum load was increased stepwise from 2000 up to 6000 N,

with 1 million cycles performed at each load level under the given conditions. In this case, failure occurred at the upper threaded section of the Nimonic fixture only at a maximum load of 6000 N after 430,000 cycles (Fig. 4c, specimen F02-01 in Table 2).

In the fourth test (specimen F03-01), the temperature was increased from 500 to 700 °C, and the maximum load was increased in 500 N increments, from 1000 to 4000 N, compared to the previous tests. At each maximum load level, 1 million cycles were applied. Under this testing regime, a seizure occurred at the composite–test fixture interface at a maximum load of 4000 N after 800,000 cycles (Fig. 4d, specimen F03-01 in Table 2).

Notably, interesting results were obtained dur-

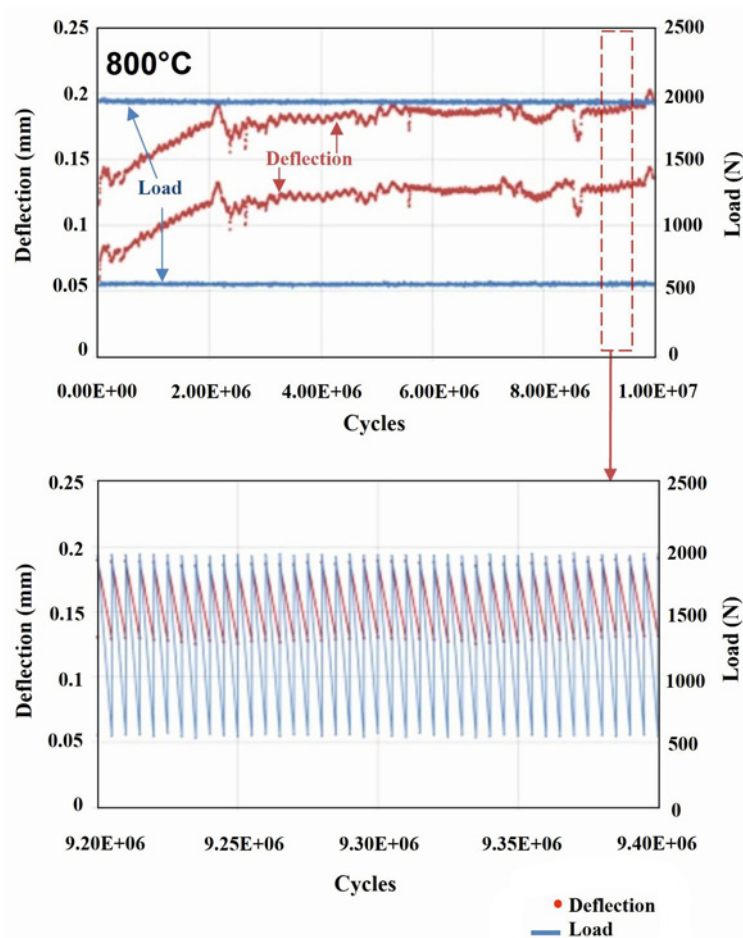


Fig. 5. Fatigue loading profile recorded at 800°C for specimen F03-02, with a minimum load of 500 N and a maximum load of 2000 N. The lower image provides a detailed view (magnified inset).

ing testing of the fifth specimen (specimen F03-02), which was exposed to 800°C and a constant maximum load of 2000 N throughout the test. No mechanical damage was observed even after 10 million cycles (Fig. 4e, specimen F03-02 in Table 2). The continuous dynamic loading profile and deflection performance for specimen F03-02 are graphically plotted in Fig. 5, where decimal values on the axes display high-precision measurements. The profile maps the displacement stability throughout the 10 million cycles, proving that the structural integrity of the assembly was perfectly maintained under high-temperature cyclic loading. These results indicate that the maximum load level has a more significant influence on specimen failure than the cumulative number of cycles applied at different load levels. For specific applications, such high loads are not required; for example, fatigue testing of exhaust valves in gasoline engines is typically performed within a load range of 500 to 2000 N.

3.2. Microstructure

Initially, after infiltration, the matrix consisted of the phases NiAl_3 , Ni_2Al_3 , and residual pure aluminum (as reported in the previously published baseline work [10]). After fatigue testing, specimens were extracted from sample F3-02 for microstructural analysis. During fatigue loading, phases NiAl_3 , Ni_2Al_3 and residual aluminum completely transformed into the more stable intermetallic phases NiAl and Ni_3Al (Fig. 6). This significant phase evolution observed in the bulk composite matrix after the fatigue test provides the explanation for the elevated thermodynamic stability of the material. The combined effect of cyclic mechanical energy and continuous exposure to 800°C accelerates the interdiffusion kinetics, shifting the multiphase system toward thermodynamic equilibrium as described by the interdiffusion growth models [12]. This final B2-ordered NiAl matrix layout guarantees excellent macrostructural stability and prevents any further sub-surface degradation during long-term high-temperature operations [6, 13].

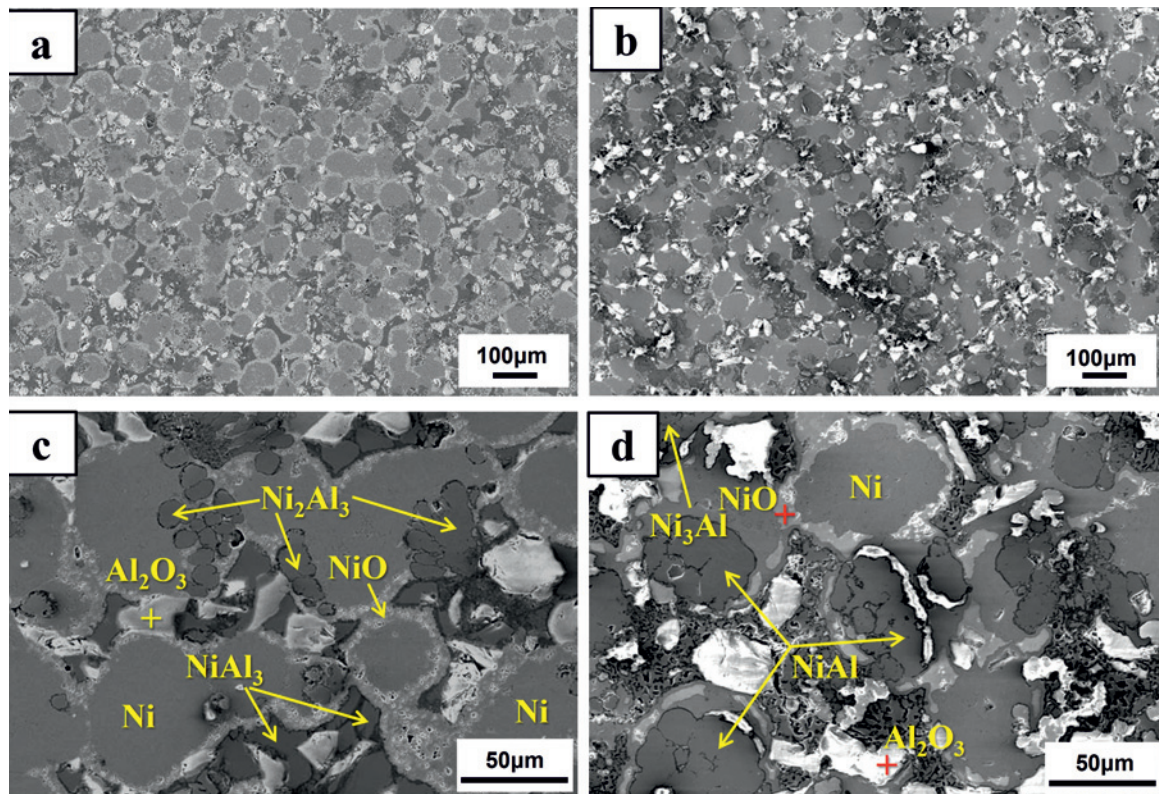


Fig. 6. SEM micrographs of the bulk Ni-Al-Al₂O₃ composite showing the microstructure before the test (a), (c) and the phase transformation into stable NiAl and Ni₃Al phases after 10 million fatigue cycles at 800°C (b), (d).

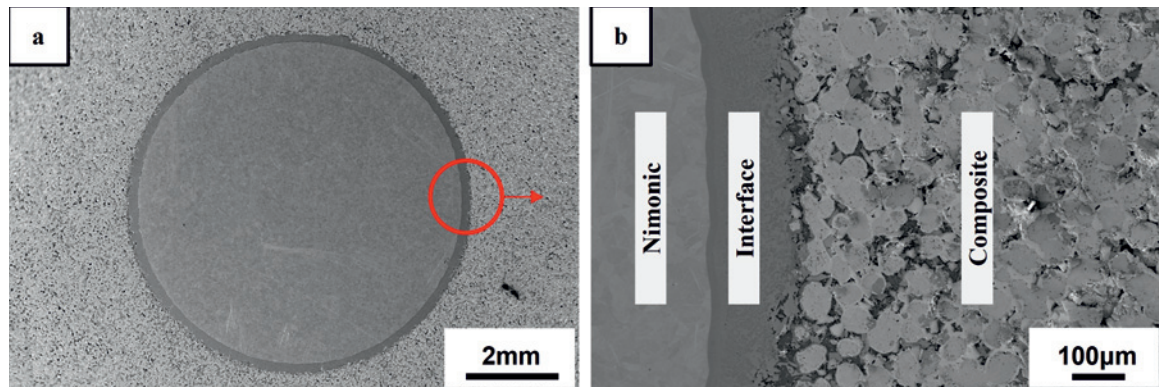


Fig. 7. SEM micrograph of the initial interface between the Nimonic 80A core, transition layer zone, and the Ni-Al-Al₂O₃ composite head prior to fatigue testing, highlighting the 150 µm reactive transition zone.

4. Conclusions

In this study, the high-temperature functionality and structural stability of a novel Ni-Al-Al₂O₃ metal-ceramic composite joined with a Nimonic 80A superalloy stem were successfully verified via systematic fatigue testing at 800°C. The experimental findings and microstructural analyses can be concluded as follows:

- *Interfacial integrity:* The metallurgical bond between the composite head and the Nimonic 80A stem exhibited exceptional fatigue resistance. Under a constant maximum shear force of 2000 N at 800°C and

a frequency of 20 Hz, the joined system successfully withstood 10 million cycles. No macroscopic failures or delamination were observed at the joint, validating the high reliability of the 150 µm reactive transition zone (Fig. 7) and the robust chemical gradient formed by Cr diffusion (Fig. 8) during infiltration.

- *Phase evolution of the composite bulk:* Microstructural characterization of the bulk composite material (Fig. 6) confirmed that the matrix undergoes a complete phase transformation under service-like thermal and mechanical loading. The initial post-infiltration matrix, containing metastable NiAl₃ and

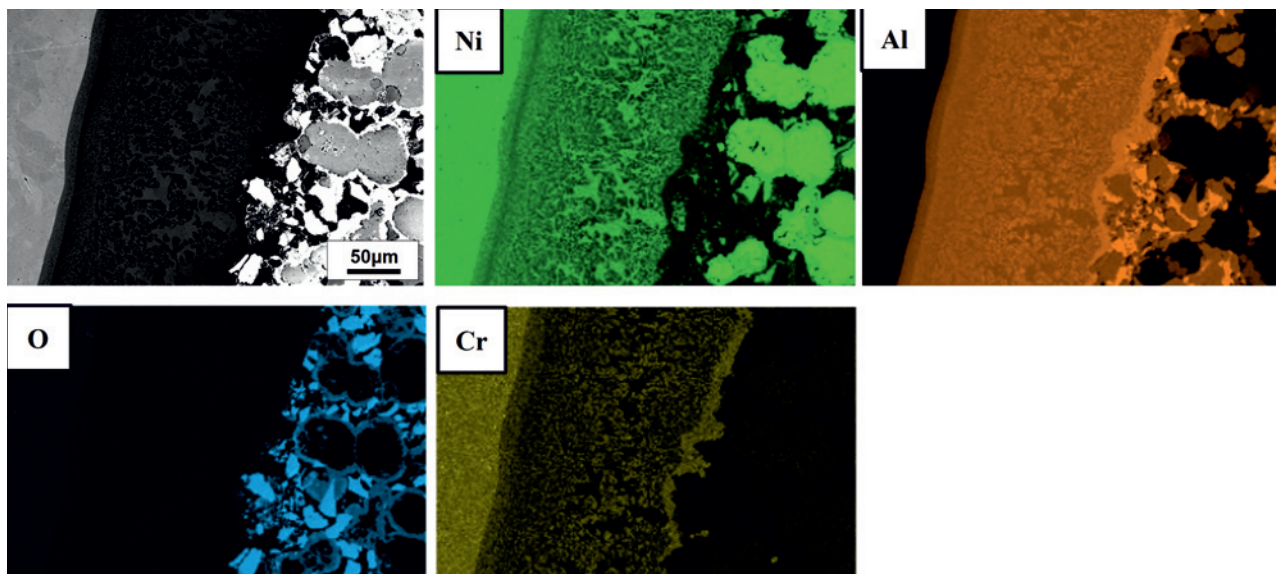


Fig. 8. SEM-EDS analysis of phase layer growth and multi-component interdiffusion mechanisms in the nickel-aluminum system under high-temperature exposure prior to testing, defining the Nimonic base, transition interlayer, and outer composite region.

Ni_2Al_3 phases alongside residual aluminum, fully transformed during the 10 million cycles into the thermodynamically stable intermetallic phases NiAl and Ni_3Al .

– *Processing implications:* The results demonstrate that reactive synthesis via gas-pressure infiltration allows for a controlled but initially metastable phase composition. To guarantee dimensional stability and optimal performance prior to component service, the structure should be stabilized by a designated post-infiltration heat treatment at temperatures exceeding the maximum expected operating temperature of the engine component.

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