Nondestructive measurement of the mechanical properties of graphene nanoplatelets reinforced nickel aluminium bronze composites

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ARTICLE INFO

Keywords:
Nondestructive measurements
Nanoindentation
Graphene nanoplatelets
Spark plasma sintering
Nickel aluminium bronze

ABSTRACT

Nanoindentation is a viable method to assess the mechanical properties of developed alloys and composites at the nanometer scale without hampering the microstructure and integrity of materials. In this study, nondestructive measurement was conducted on spark plasma sintered nickel aluminium bronze (NAB), and graphene nanoplatelets (1, 2, 3 wt.%) reinforced NAB composites using the nanoindentation technique. The nondestructive measurements were conducted under loads of 50 mN and 100 mN to assess the nanohardness and reduced elastic modulus of the fabricated NAB alloy and composites. Further investigations were carried to evaluate the elastic recovery index, plasticity index, the nanohardness and reduced modulus ratio, and the yield pressure to reveal the nanomechanical responses of the fabricated materials. Scanning electron microscopy was used to analyze and reveal the dispersibility of the graphene nanoplatelets (GNP) in the NAB matrix. The nondestructive measurements showed that the nanohardness, reduced elastic modulus, yield pressure, resistance to elastic strain to failure and the elastic recovery index improved with the presence and increase in the concentration of GNP in the NAB matrix. The reduced elastic modulus and nanohardness values range from 34.2 – 43.0 GPa and 4407.2 – 6598.8 MPa respectively, which declined with nanoindentation loads. The fabricated NAB alloy experienced the maximum plastic deformation and least resistance to impact loading.

1. Introduction

The demand for lightweight and advanced materials for the replacement of conventional monolithic materials has been on the increase in recent years. This has engendered the fabrication of advanced alloy systems and composites with improved properties using advanced manufacturing techniques for structural, aerospace, biomedical, automobile and marine applications.

Over the past decades, Nickel aluminium bronze (NAB) has been widely used for the production of diverse components for aerospace, automobile, marine and oil and gas industries owing to their mechanical, aesthetic, resistance to wear and galling as well as good corrosion resistance [1, 2]. Also, NAB alloys are heat treatable, weldable, castable, and their superior corrosion resistance is attributed to the thick oxide layer that is usually formed on their surfaces. NAB alloys can retain their mechanical properties by not undergoing brittle transition at lower temperatures; that is why they are widely employed in cryogenic environments in the marine and oil and gas industries [3]. They are extensively used to fabricate valves, propellers, aircraft landing gear, winch gear, wind flap bearings, and window frames, to mention but a few. The widespread applications of the NAB alloy system may be ascribed to the chemical composition and the complex microstructure, which results in the improved properties of the alloy [4].

A typical NAB alloy system majorly contains copper (Cu) and various alloying elements, which are added for a specific function. For instance, aluminium (Al) between 8-13% is usually added as strengthening metal to improve the hardness and wear resistance, while nickel (Ni) between 1-7% enhances the alloy's corrosion resistance and mechanical strength. Other alloying elements such as manganese (Mn) are usually added as deoxidants and promote the alloy's fluidity during casting [2]. As-cast NAB alloy comprises complex microstructures, which contain alpha, retained beta and intermetallic phases. Also, precipitates in the form of kappa (ksi-iv) phases usually occur in the alloy system [5]. Each of the precipitates can be differentiated by their location, morphology and dispersion in the microstructure of the alloy, and their presence can...
influence the fatigue, mechanical and corrosion behaviours of the alloy [6, 7].

Despite the remarkable and promising properties and extensive applications of NAB alloys, past studies have highlighted the complicated microstructures of the alloy system, which has a relationship with the various alloying elements and the numerous phase transformations that occur during the production of the alloy. Also, the alloy tends to lose its good mechanical properties in sulphide environments and at elevated temperatures. In order to improve the properties of the NAB alloy system, various heat treatment processes have been employed such as stress relieving, annealing, quenching and tempering or ageing to relieve internal stresses formed in the alloy during production, improve the ductility, increase the hardness and enhance the corrosion resistance [2]. Even at the advancement in materials development, incorporating second phase particles into the NAB alloy system to improve the properties has not yet been explored, especially using reinforcements such as carbon nanotubes (CNT) and graphene nanoplatelets (GNP).

In recent times, GNP has been massively used to augment the properties of various metals and alloys for diverse engineering applications owing to its excellent mechanical, thermal, corrosion resistance and physical properties [8, 9]. GNPs are 2D layers of graphene sheets that are preferable as reinforcement materials than CNT due to their larger surface area, ease of dispersion and lesser tendencies to break during integration into matrix materials [10, 11]. It has exclusive properties; elastic modulus in the range of 1TPa, thermal conductivity of 5000 W/mK and surface area of 2630 m²/g [12]. These properties are usually transferred into matrix materials when utilized as a reinforcement phase.

In the past years, various NAB alloys have been fabricated using conventional casting techniques. However, due to the complexity of the phases, segregations of phases in the alloy system, and the formation of different precipitates, post-processing techniques such as heat treatment is desirable in achieving desired properties. In recent times, research advances in materials development have favoured the application of powder metallurgy techniques such as spark plasma sintering (SPS) in fabricating NAB systems with finer microstructures and limited grain segregations. Spark plasma sintering is an improved consolidation technique that employed uniaxial load to compact powder materials at a lesser time, lower temperature and minimizes grain growth during consolidation [13]. Past works have emphasized the advantages of the application of SPS over other conventional materials processing techniques in the fabrication of alloys and metal matrix composites which are quite enormous [13, 14].

Apart from fabricating materials with desired microstructure, the testing of the mechanical properties is also crucial in ascertaining the behaviour of materials in real-life conditions. Over the years, diverse destructive and micromechanical testing techniques such as tensile, hardness, impact, to mention but a few, have been adequately employed in ascertaining the mechanical properties of NAB alloys. However, due to technological advancement and the publication of Oliver and Pharr in the early 1990s has revealed the importance of probing the mechanical behaviours of materials at a nanoscale range using the nanoindentation technique [15].

Nanoindentation technique has been effectively used to probe the mechanical properties of various materials, namely ceramics, polymers, alloys, and composites, without hampering the material’s microstructural orientation [16]. It can be utilized in probing the nanomechanical behaviours such as reduced elastic modulus, nanohardness, creep strength, friction toughness, plasticity index, anti-wear and elastic-plastic behaviours of various small scale materials [17, 18]. In recent years, nanoindentation has been extensively used to measure the mechanical properties of thin films and coating of various materials and small samples without prior machining of the samples to any particular shape and size [19]. Although the nanoindentation technique is a sensitive measuring method, it can measure the mechanical properties of materials within a limited time and gives precise readings at the nanoscale [20]. Maja et al. [21] utilized this measuring technique to probe the mechanical properties of titanium-based composites reinforced with titanium nitride, where nanohardness, elastic modulus and elastic-plastic behaviour of the composites was reported.

Similarly, Okoro et al. [18] reported the plasticity index, yield pressure, elastic strain recovery, nanohardness of titanium-based nano-composites reinforced with CNT from the nanoindentation measurements. Despite the numerous research conducted in the past on the mechanical properties of NAB alloy and its composites, to the best of our knowledge, there are no research findings on the nanomechanical properties of spark plasma sintered NAB alloy and its composites. Hence, in this research, nondestructive measurements were conducted using the nanoindentation technique to investigate the nanomechanical properties of GNP reinforced NAB composites developed by the SPS technique.

2. Materials and methods

The starting materials used in this study and the method of synthesis of the alloy and composites are explained in this section. The starting metallic powders were mixed homogeneously using an effective powder mixing technique. Subsequently, the mixed powders were then consolidated via spark plasma sintering. In order to ascertain the nanomechanical properties of the composites, nanoindentation was used to probe the developed alloy and composites.

2.1. Synthesis and fabrication of NAB and GNP-NAB composites

The NAB alloy was developed from the elemental powders (Cu, Al & Ni). In a bid to produce the NAB powder, the elemental powders comprising Cu (80 wt.%), Al (10 wt.%) and Ni (4 wt.%) were mixed homogeneously using the shift-speed milling (SSBM) technique as reported in our previous study [22]. Similarly, the GNP (0.5, 1.0 and 1.5 wt.%) was dispersed in the NAB powders using the SSBM technique. The mixing was conducted by milling the starting powders for 8 h at a speed of 150 rpm and a ball to powders ratio (BPR) with 5mm alumina balls of 10:1 using a low-speed ball mill (Retch PM 100). Subsequently, the pre-mixed powders were transferred into a high-speed ball mill (Retch 400 PM) for further milling at a speed of 100 rpm, for 2 h using a BPR of 10:1 to achieve uniformity and even dispersion of the GNP in the NAB powders.

The admixed powders were then cold compacted at a load of 10kN in a 20 mm graphite die to achieve green strength. Afterwards, the compacted powder was transferred into the SPS (model HHPD-25, FCT GmbH Germany) for consolidation. The sintering was conducted at a temperature of 800 °C, using a holding time of 5 min, a heating rate of 100 °C/min and a pressure of 50 MPa. The aforementioned sintering parameters were used to fabricate the NAB alloy and GNP-NAB composite grades. After the sintering, the consolidated alloy and composite grades were sandblasted to remove graphite contaminants used during the sintering process and the relative densities were measured in agreement with ASTM B962 [23].

2.2. Microstructural study of the developed NAB and GNP-NAB composites

Prior to the microstructural examination of the NAB alloy and the composites, the fabricated materials were ground using silica carbide papers (P800 to 1200 grit size) and polished using colloidal paste on polishing cloths (3 to 1µ) to achieve a smooth and mirror-like finish. The polished samples were then viewed under a scanning electron microscope (Carl Zeiss Sigma FESEM) to ascertain the microstructural features of the materials.

2.3. Nanoindentation of the fabricated NAB and GNP-NAB composites

The nanomechanical properties of the fabricated NAB alloy and composites were investigated using an ultra nanoindenter (UNHT),...
Switzerland, in accordance with ISO 14577 [24]. The nanoindenter uses a diamond Berkovich (3 sided) indenter to probe the polished surface of materials to reveal the elastic-plastic behaviour of indented materials at a nanoscale range. Prior to the nanoindentation test, the UNHT equipment was calibrated using a fused silica reference sample to enhance the accuracy of the test results. The nanoindentation test was carried out with loads of 50 and 100 mN using a loading and unloading rate of 10 mN/min. In order to eliminate the viscoelastic effect and determine the time-dependent deformation of the samples, a pause of 1000s and over 15 indentations on each sample were done in this study. The basic nanomechanical properties that were investigated in this study are nanohardness, reduced elastic modulus, elastic-plastic behaviours, elastic recovery index, plasticity index, elastic strain resistance and yield pressure at the ultimate load and these properties were evaluated based on the Oliver and Pharr method [25].

The nanohardness was evaluated from the ratio of the ultimate load ($F_{\text{max}}$) to the contact area ($A_c$) of the projected sample at the maximum load, which is shown in Eq. (1) below [25]. In contrast, the reduced elastic modulus was derived from the unloading area of the load-displacement curve during nanoindentation.

$$H_N = \frac{F_{\text{max}}}{A_c}$$  \hspace{1cm} (1)

In Figure 1, the compliance curve, $h_c$, represent the depth of the contact circle, while the reduced elastic modulus and nanohardness are calculated from the slope of the unloading section of the load-displacement curve. Also, $h_r$ is the depth of the residual impression, and $h_e$ is the displacement relating to the elastic recovery of the specimen during unloading. The contact stiffness is dependent on the indented area ($A$) at maximum load, and the reduced elastic modulus ($E_r$). It is expressed as the relation in Eq. (2) below [25]:

$$S = \frac{2}{\sqrt{\pi}} E_r \sqrt{A}$$ \hspace{1cm} (2)

The reduced elastic modulus ($E_r$) of a sample during nanoindentation can be calculated using the expression in Eq. (3); the expression shows the relationship of the Poisson’s ratio for the sample ($\nu_s$) and the indenter ($\nu_i$) and the elastic modulus of the sample $E_s$ and the indenter $E_i$ respectively [26].

$$\frac{1}{E_r} = \frac{1 - \nu_i^2}{E_i} + \frac{1 - \nu_s^2}{E_s}$$ \hspace{1cm} (3)

In order to calculate the total work done by the indenter during nanoindentation, the work done expression can be extrapolated from the displacement of the indenter with respect to the load during nanoindentation. The displacement of the indenter and the load is in accordance with power law [27], which is expressed in Eqs. (4) and (5).

$$F = a \cdot h^m$$ \hspace{1cm} (4)

$$F = a (h - h_f)^m$$ \hspace{1cm} (5)

From the expression in Eqs. (4) and (5), $F$ is the indentation load, $h$ is the instantaneous indenter displacement, $h_f$ is the final indentation depth of residual impression and $m$, $a$, $m_1$, $a_1$ are the empirical fitting parameters.
parameters. The value for the ultimate depth ($h_{\text{max}}$) at the maximum load $F_{\text{max}}$ can be derived by summing up the contact depth ($h_c$) and the elastic surface displacement ($h_e$) [20, 25] as expressed in Eq. (6).

$$h_{\text{max}} = h_c + h_e$$  

(6)

The total work done by the indenter ($W_t$) on the sample during nanoindentation was derived from the expressions in Eqs. (4) and (5) by Bao et al. [27]. The total work done can be used to calculate the elastic recovery index (ERI) ($W_e / W_t$) of the samples during nanoindentation, which specifies the energy dissipated from a sample after the removal of an indentation load and it demonstrates the impact loading resistance of an indented sample [28]. Also, the total work done by the indenter can be used to calculate the plasticity index (PI) ($W_p / W_t$), which also give information about the mechanical behaviours of an indented sample during nanoindentation, and it gives the intrinsic plasticity of a sample [29, 30]. The total work done by the indenter during indentation can be expressed as in Eq. (7).

$$W_t = W_e + W_p$$  

(7)

where $W_e$ and $W_p$ represent the elastic energy and plastic energy respectively, and these can be calculated from the area under the load-displacement nanoindentation curve as shown in Figure 1.

3. Results and discussion

3.1. Microstructural analysis of the fabricated NAB and NAB based composites

The microstructural features of the starting powders are presented in Figure 2 (a-d). From the Figure of the starting powders, it was observed that the elemental metallic powders depict spherical particles, and the Cu powders have an average particle size of ~85.7 μm. In contrast, the Ni and Al powders comprise spherical particles with average sizes of ~29.5 and ~15.4 μm respectively. Also, the GNP used as the reinforcement phase shows flake-like morphology, and it consists of 5 μm particle size and surface areas of 50–80 m²/g. Similarly, the SEM/EDS images of the fabricated NAB and NAB based composites which comprised 1, 2 and 3 weight percent of GNP is presented in Figure 3 (a-d). Figure 3(a) shows the morphology of the fabricated NAB alloy, which reveals the presence of nickel rich phases surrounding the Cu phases. It also discloses the densely packed particles of the fabricated alloy, showing the effectiveness of the sintering process and parameters used in this study. The SEM image in Figure 3(b) presents the fabricated NAB based composite, which contains 1 wt.% GNP, and it reveals the presence of GNP situated within the interstices of the NAB alloy. The GNP segregation behaviours into the interstices of the NAB alloy could be ascribed to their smaller particle sizes, lower densities, volume and difference in physical properties from the NAB alloy. The presence of GNP in the interstices of the NAB alloy could assist in preventing the distortion of the alloy in-service condition, especially at elevated temperatures since graphite has the tendencies of retarding the swelling of copper alloys at elevated temperatures [31]. Similarly, Figure 3(c) and (d) shows the microstructural features of the fabricated NAB composites reinforced with 2 and 3wt.% GNP respectively. From the microstructural features, it was observed that the GNP are also situated at the interstices of the samples in Figure 3(c) and (d), some dot-like carbon residues were observed on the micrograph in Figure 3(d).

Figure 3. SEM images of the fabricated NAB (a), 1 wt.% GNP-NAB (b), 2 wt.% GNP-NAB (c) and 3wt.% GNP-NAB (d) Composites alongside EDS, showing the composition of the nickel rich NAB alloy grains (e) and Composite grains (f).
and this could be ascribed to the higher weight fraction of GNP in the composite composition that resulted in the precipitation of the GNP in the form of carbon. Also, Figures 3 (e) and (f) show the EDS results of the sintered alloy and composites. In the EDS result shown in Figure 3 (e), the nickel rich region on the alloy is revealed and it confirms the presence of other alloying elements in the same region (Cu & Al). Similarly, Figure 3 (f), confirms the presence of carbon within the copper-aluminum-nickel matrix, which also reveals the presence of GNPs in the composite system.

3.2. Nanomechanical properties of the fabricated NAB and NAB based composites

The basic nanomechanical properties that were tested on the fabricated materials using the nanoindentation technique are nanohardness, reduced elastic modulus, elastic recovery index and plasticity index. Furthermore, the load-displacement behaviour and the displacement of the materials with respect to indentation time during nanoindentation was assessed.

3.2.1. Displacement-time behaviour of the fabricated NAB and NAB based composites

The displacement with respect to time plots of the fabricated materials under the influence of 50 mN and 100 mN are presented in Figure 4 (a) and (b), respectively. It was observed that the fabricated NAB alloy and the NAB based composites showed enormous plastic deformation during the nanoindentation test. In Figure 4 (a), the NAB alloy depicted the maximum plastic deformation due to the indentation depth (1004.8 nm) experienced. While the 3wt.% GNP-NAB composite grade showed the least plastic deformation (939.4 nm), followed by 2wt.% GNP-NAB (943 nm) and 1wt.% GNP-NAB (970.8 nm). The depth-time plot also shows that the indentation depth decreased with the increase in GNP concentration which implies that the presence of GNP enhances the mechanical strength of the fabricated composites. The enhancement in mechanical strength could be ascribed to the pinning effect and dispersion strengthening mechanism resulting from the nanoplatelets in the NAB matrix [34]. When the indentation load was increased to 100 mN, the fabricated alloy and composites also depict similar behaviour as with lower load, and the result is presented in Figure 4 (b). The sintered NAB alloy also displayed the highest indentation depth (1406.9 nm) under 100 mN while 3wt.% GNP-NAB composite grade showed the least indentation depth (1263.7 nm). The 1wt.% GNP-NAB composite grade experienced the highest indentation depth (1286.8 nm) among the fabricated composites and was accompanied by the 2wt.% GNP-NAB grade.

3.2.2. Nanohardness and reduced elastic modulus of the fabricated NAB and NAB based composites

In a bid to ascertain the structural performance of the fabricated alloy and composites, the materials were subjected to indentation loads of 50 mN and 100 mN to reveal the nanohardness and reduced elastic modulus. The results of the nanohardness and reduced elastic modulus of the fabricated materials are presented in Figure 5 (a and b). From the nanohardness results in Figure 5 (a), it was observed that the fabricated composites exhibited significant resistance to the surface indentation under the two tested loads (50 mN and 100 mN). The nanohardness improved with the increase in GNP concentration in the NAB matrix; the fabricated composite grade (3wt.% GNP-NAB) with the highest GNP content exhibited the maximum nanohardness values of 6598.8 MPa and 6338 MPa under the influence of 50 mN and 100 mN indentation loads respectively. Meanwhile, the fabricated NAB alloy displayed the lowest nanohardness values of 5624.3 MPa and 4407.2 MPa when subjected to indentation loads of 50 mN and 100 mN respectively. Correspondingly, 2wt.% GNP-NAB composite grade displayed the nanohardness values of 6173.2 MPa and 5971.5 MPa under loads of 50 mN and 100 mN respectively. In comparison, 1wt.% GNP-NAB composite grade showed nanohardness values of 5796.5 MPa and 5466.9 MPa under indentation loads of 50 mN and 100 mN. From the nanohardness response of the fabricated materials, it could be deduced that the
nanohardness improved with the increase in GNP content, and it declines with the increase in indentation loads. The improvement in nanohardness resulting from the addition of GNP in the NAB matrix could be ascribed to the load transfer from the matrix to the stiffer reinforcement. Also, the increase in nanohardness value could result from the dispersion strengthening provided by the GNP in the NAB matrix [35].

The reduced elastic modulus results, which explain the response of the fabricated material to linear compression loading, is presented in Figure 5(b). It was observed that the fabricated composites experienced significant improvement in elastic modulus due to the increase in GNP content. However, the increase in elastic modulus declined with a higher indentation load. A similar trend to the nanohardness results was recorded as 3wt.% GNP-NAB composite grade displayed the maximum reduced elastic modulus (43.0 GPa and 41.6 GPa), followed by 2wt.% GNP-NAB (41.6 GPa and 40.7 GPa) and 1wt.% GNP-NAB (39.1 GPa and 38.0 GPa) under the indentation loads of 50 mN and 100 mN respectively. However, the fabricated NAB alloy displayed the lowest reduced modulus values of 36.7 GPa and 34.2 GPa under the indentation loads of 50 mN and 100 mN. The improvement in reduced elastic modulus could be ascribed to the dispersion strengthening mechanism and the load transfer mechanism from the matrix to the reinforcement.

3.2.3. Anti-wear and nanomechanical response of the fabricated NAB and NAB based composites

Further analysis was conducted on the nanoindentation results to understand the elastic and plastic behaviours as well as the anti-wear response of the fabricated materials under 100 mN load. The nanomechanical (elastic and plastic) and anti-wear response of the fabricated materials were assessed from the elastic recovery index ($W_t/W_i$), plasticity index ($W_p/C_14$), the nanohardness and reduced modulus ratio ($H/E_r$) as well as the yield pressure ($H^3/E_r^2$). The elastic recovery index is usually employed to ascertain the energy dissipated by the material when subjected to indentation loads. In contrast, the plasticity index reveals the inherent plastic response of materials during nanoindentation [36]. The resistance of the materials to elastic strain to failure at the nanoscale is ascertained using the nanohardness, and reduced elastic ratio, while the resistance to plastic deformation and wear during indentation is assessed using the yield pressure [37, 38, 39].

From the elastic strain resistance and yield pressure results in Figure 6(a), it was observed that the elastic strain to fracture values and the resistance to plastic deformation and wear of the fabricated materials improved with GNP concentration. The composite grade (3wt.% GNP-NAB) with the highest GNP content exhibited the maximum resistance to plastic deformation and wear during the nanoindentation measurements and 2wt.% GNP-NAB and 1wt accompanied this trend. However, the fabricated NAB alloy showed the least resistance to plastic deformation and wear during the nanoindentation measurement.

Similarly, from the elastic recovery index results in Figure 6(b), it was observed that the composites grades dissipated higher energy during nanoindentation measurements which increased with the concentration of the GNP. However, the fabricated NAB alloy showed the highest plasticity index, implying that the alloy undergoes the maximum plastic deformation during the nanoindentation measurement. It could be deduced from the plasticity index results in Figure 6(b) that the increase in GNP concentration resulted in the resistance of the composites to plastic deformation. The results obtained in this study are similar to the trends reported in nanoindentation studies of various alloys and metal matrix composites [18, 40].

4. Conclusion

In a quest to understand the nanomechanical response of spark plasma sintered nickel aluminium bronze and GNP reinforced nickel aluminium bronze-based composites, nondestructive measurements were conducted using nanoindentation technique in this study, and the following conclusions were drawn:

1. The nanohardness and reduced elastic modulus of the fabricated NAB alloy and NAB based composites significantly declined with the increase in nanoindentation load. The nanohardness and reduced elastic modulus values range from 4407.2-6598.8 MPa and 34.2-43.0 GPa respectively.
2. The fabricated NAB alloy displayed the maximum plastic deformation during the nanoindentation measurement.
3. The presence and increase in GNP concentration in the NAB matrix resulted in the improvement of nanohardness, reduced elastic modulus, yield pressure and elastic recovery index of the fabricated composites.

Declarations

Author contribution statement

Okoro Awasoroeghene Moses: Conceived and designed the experiments; Analyzed and interpreted the data; Wrote the paper.
Lephuthing Senzeni Sipho, Olubambi Peter Apati: Analyzed and interpreted the data; Contributed reagents, materials, analysis tools or data.
Rasiwela Livhuwani: Performed the experiments; Analyzed and interpreted the data.

Funding statement

This work was supported by the National Research Foundation of South Africa and Global Excellence and Stature of the University of Johannesburg, South Africa.
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