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# THE EFFECTS OF NICKEL ADDITIONS ON MICROSTRUCTURAL AND HARDNESS OF BALL MILLED OF AL-6WT%ZN-3WT%MG-2WT%CU ALLOYS UNDERWENT THE ARTIFICIAL AGING

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## ABSTRACT

*High-energy ball milling was employed to synthesize Al-Zn-Mg-Cu alloy (7000 series) with high weight percentage of nickel from its elemental powders via mechanical alloying mechanisms. The mixed powders underwent 15 hrs of milling time, 380 rpm speed and 12:1 balls/powder weight ratio. The milled powder samples were cold compacted and sintered thereafter. The sintered compacts which were homogenized at 450°C for 1.5 h and aged at 124 °C for 24 hrs. The milled powders and heat-treated Al alloy products were characterized via scanning electron microscopy, and energy dispersive spectroscopy. The results revealed that optimum milling time of 15 hrs has led to the formation of solid solutions of Al-Zn-Mg-Cu with Ni. The mechanical alloying mechanism allows for the attainment of Al solid solutions with amount of nickel and dopants Zn, Mg, and Cu at a concentration that exceeds their equilibrium solubility compared with melting and crystallization in conventional processes. The outcomes showed that the hardness of the sintered Al-Zn-Mg-Cu-Ni alloys compacts was enhanced following the artificial aging treatment.*

**Keywords:** Milling alloying, Al-7xxx alloys, Al-Ni compounds, Heat treatment, Hardness, SEM.

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## Contribution/ Originality

This study is one of very few studies which have investigated the possibility of manufacturing the Al–Zn-Mg-Cu-Ni intermetallic compounds by mechanical alloying mechanisms with 12 wt. % and 16 wt. % of Ni, as well as the roles of compounds of Ni in the generating of superior the microstructural and mechanical properties of the sintered Al–Zn–Mg–Cu alloys after applying the artificial aging.

## 1. INTRODUCTION

Intermetallics display an attractive combination of physical and mechanical properties. The Al-Ni gained noticeable interest and are considered as potential materials for high temperature and coating applications because of their low densities, high-melting points, high corrosion and oxidation resistance at high temperatures [1, 2]. The technological "pull" comes mainly from the needs of the aerospace industry for new high-temperature structural materials with properties that cannot be met by ceramics or by conventional superalloys. In the case of intermetallic compounds, where Al constitutes majority of the alloy, low density is an additional advantage [2, 3]. In general, conventional processing techniques such as melting and casting methods are inapplicable to the fabrication of many

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intermetallic alloys due to, for example, a large difference between the melting points of constituent elements [4]. However, this method has some disadvantages, which resulted from the large difference between the melting points of Al and Ni or connected with evaporation and oxidation [4, 5]. Alternatively, some novel processing techniques, including the Exo-Melt process [5] self-propagating high-temperature synthesis [6] friction stir processing [7, 8] and mechanical alloying (MA) [9] have been used to produce nickel aluminide intermetallic compounds. During the MA process, alloys are formed by solid-state reaction.

Therefore, the MA technique overcomes problems, such as the large difference in melting points of the alloying components and evaporation or segregation that could occur during melting and casting. MA is a dry powder processing technique and has been used to synthesize nanostructured intermetallic compounds with ductile behavior. This technique involves repeated cold welding and fracturing of powder particles in a high-energy ball mill [10]. Effective parameters in MA are time, speed of milling, ball-to-powder weight ratio, and atmosphere [10, 11].

Naeem, et al. [12] found that formation Al-Ni intermetallics through Al-Zn-Mg-Cu alloy after performed 5 hrs of MA. As well so many Al-Ni compounds were formed in Al-compacts samples underwent the sintering process. Naeem, et al. [13] investigated that intermetallic phases of Al-Ni formed within Al-Zn-Mg-Cu-Ni alloy produced by ball milling after 12 hrs of MA.

Mohammed, et al. [14]showed that after 15 hrs of mechanical alloying processing for the milled alloy (Al-5.5Zn-2.5%Mg-1.5%Cu) forming various compounds of Ni-rich particulates through the Al-matrix additionally good mechanical properties. In the present study, the producing synthesized of the Al-Ni intermetallic sby mechanical alloying with 12 wt. % and 16 wt. % of nickel, as well as their roles on microstructural and hardness properties of the Al-Zn-Mg-Cu alloys after carrying out the sintering the artificial aging. The microstructural, structural, and microanalysis evolutions occurring in the MA process were also investigated.

## 2. EXPERIMENTAL PROCEDURE

### 2.1. Synthesis of Alloy Powder

To synthesize Al milled powders using mechanical alloying process, the elemental powder precursors of Al, Zn, Mg, Cu, and Ni (supplied by MERCK KGaA,Germany) were used as the raw materials for the preparation of three types of milled powders, namely, Alloy-I (Al- 6% Zn- 3% Mg- 2% Cu), Alloy-II (Al- 6%Zn- 3%Mg- 2% Cu-12% Ni) and Alloy-III (Al- 6%Zn- 3%Mg- 2% Cu- 16% Ni). All compositions are expressed in weight percentage (wt. %). Table 1 lists the purity levels and the particulate sizes of the raw materials. Particles size distributions of the powders were analyzed by laser diffraction technique using Malvern Instruments Mastersizer 2000.

**Table-1.** Specifications of Materials

| Powder | Description | Average Size D50 of Particulates | Purity (%) |
|---|---|---|---|
| Al | Flake | D50 of 51 | 98.00 |
| Zn | Rounded | D50 of 18 | 96.00 |
| Mg | Rounded | D50 of 115 | 98.00 |
| Cu | Irregular | D50 of 39 | 99.50 |
| Ni | Rounded | D50 0f 11 | 99.50 |

Ball milling (BM) of the powder was conducted in a planetary high-energy ball mill (Fritsch model Pulverisette 5) under argon atmosphere using stainless steel balls of 10 mm to 20 mm diameter. The balls/powder weight ratio was 12:1 and the rotation speed was 380 rpm. MA process for the powder mixture was completed at milling time of approximately 15 hrs. Ball milling experiments were periodically halted every 15 min and then resumed for 45 min to prevent a significant increase in temperature, more detailed of the MA process in the present study [12-14].

### 2.2. Mixture Compaction, Sintering and Heat Treatments

Consolidated bulk products were prepared by cold pressing the milled Al mixtures at 600 MPa under uniaxial load. Subsequently, green compacts were sintered under argon atmosphere for 60 min. at 600 °C and a heating rate of 25 °C/min. The sintered compacts underwent homogenization treatment at 450 °C for 90 min., followed by immediate quenching in cold water. Then the compacts were the artificial aged at 124 °C for 24 h and then also quenching in cold-water was performed.

### 2.3. Characterization

The specimens were ground and polished according to ASTM E3-01. Scanning electron microscopy (SEM) was performed using JEOL JSM-6460LA coupled with an energy-dispersive X-ray spectroscope (EDS) to analyze the microstructure features. The Vickers hardness was evaluated on the compacts according to the ASTM E92-82 standard. The compacts were polished prior to Hv measurement to ensure the cleanliness of their surfaces.

## 3. RESULTS AND DISCUSSION

The initial morphologies of as-mixed and milled powder alloy-**I** are shown in Figure 1. The as-mixed powder alloy-**I** is flake-like as aluminum powder conglomerates and the remaining powders separate (Fig. 1a). After starting of milling up to 15hrs, the particles were severely deformed plastically by MA and underwent shape changes from flake-like to semispherical (Fig. 1b). In the SEM morphologies of the milled powder alloy-**II** (Fig. 2a) indicated that particles agglomerate with more diffused nickel in the soft aluminum matrix. Large aggregates were formed because of high ductility [15]. The EDS spectrum of the labeled region in Fig. 2b shows the increasing wt. % of nickel. The nanoparticles of nickel were also observed to be adherent, with scattered intensity. When MA is applied, the particulates undergo work hardening and fracture by a fatigue failure mechanism [16].

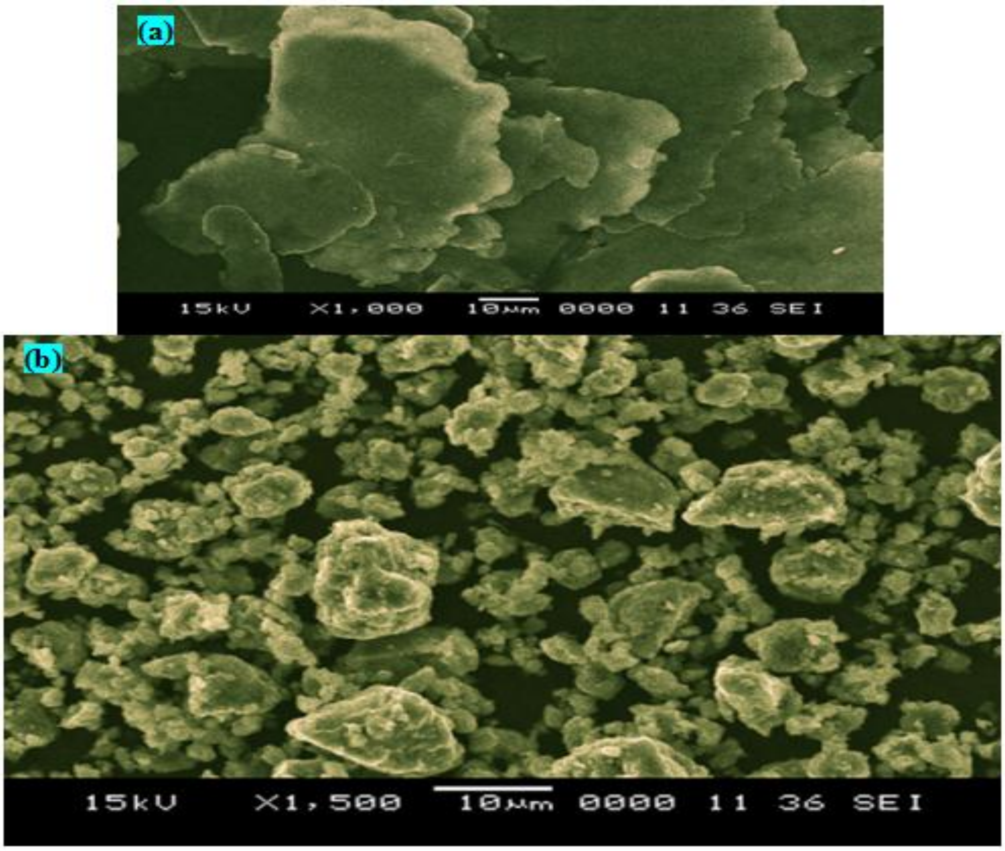

**Figure-1.** (a) and (b) show the SEM morphology of as-mixed and milled powder alloy-**I** at a milling time of 15 hrs.

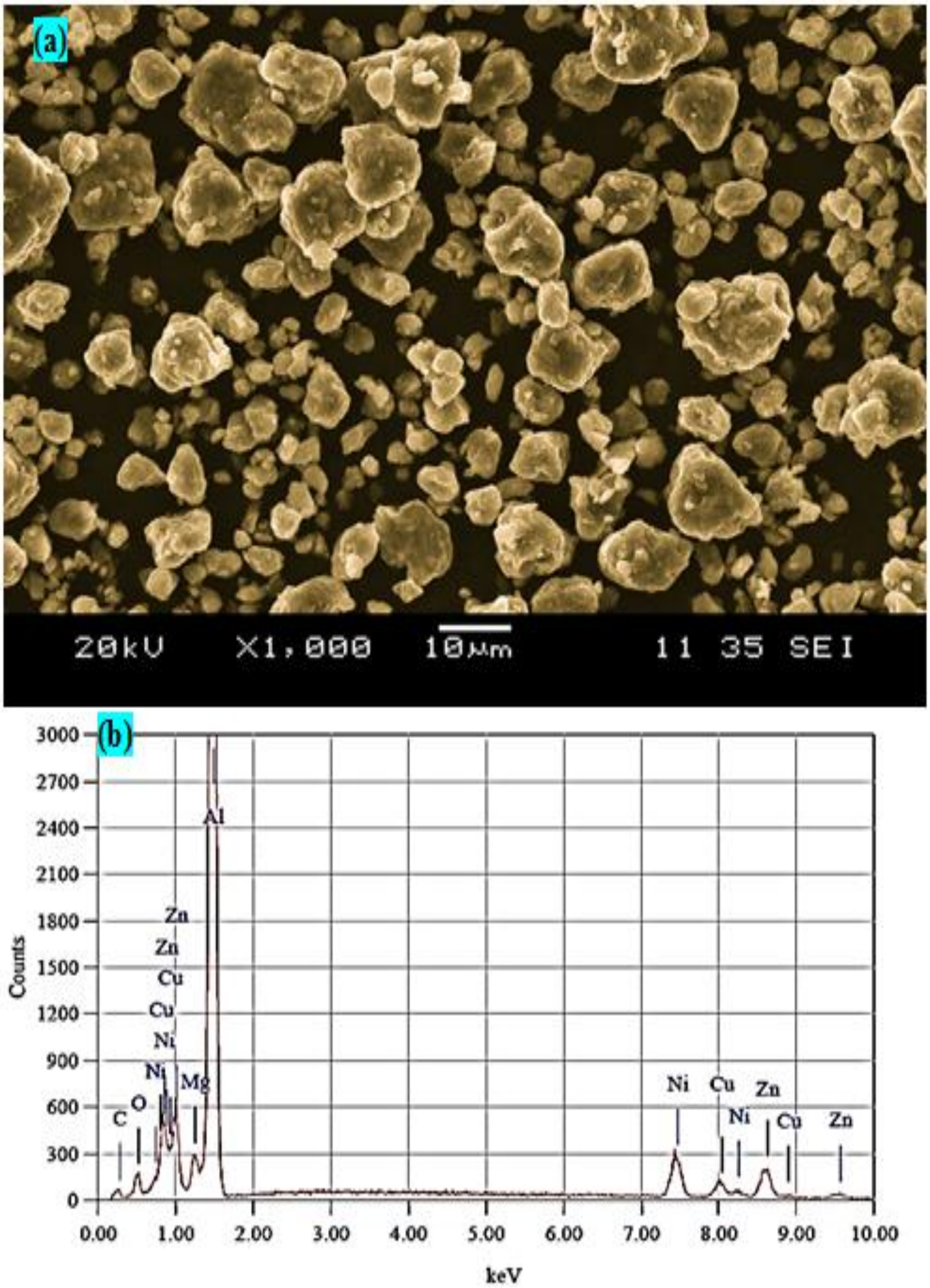

**Figure-2.** (a) show the SEM image; (b) show the EDS of the milled powder alloy-**II** at milling times of 15 hrs, respectively.

The SEM micrograph morphology of milled powder alloy-**III** after 15 hrs of ball milling, as shown in Fig. 3a, indicates a uniform distribution of the nanoparticles of Ni embedded in the Al-Zn-Mg-Cu cluster. With a long milling time, particulates were broken into smaller pieces and the mixed within matrix of alloy [17]. By contrast, fragments generated by MA continued to decrease in size in the absence of strong agglomerating forces[18].

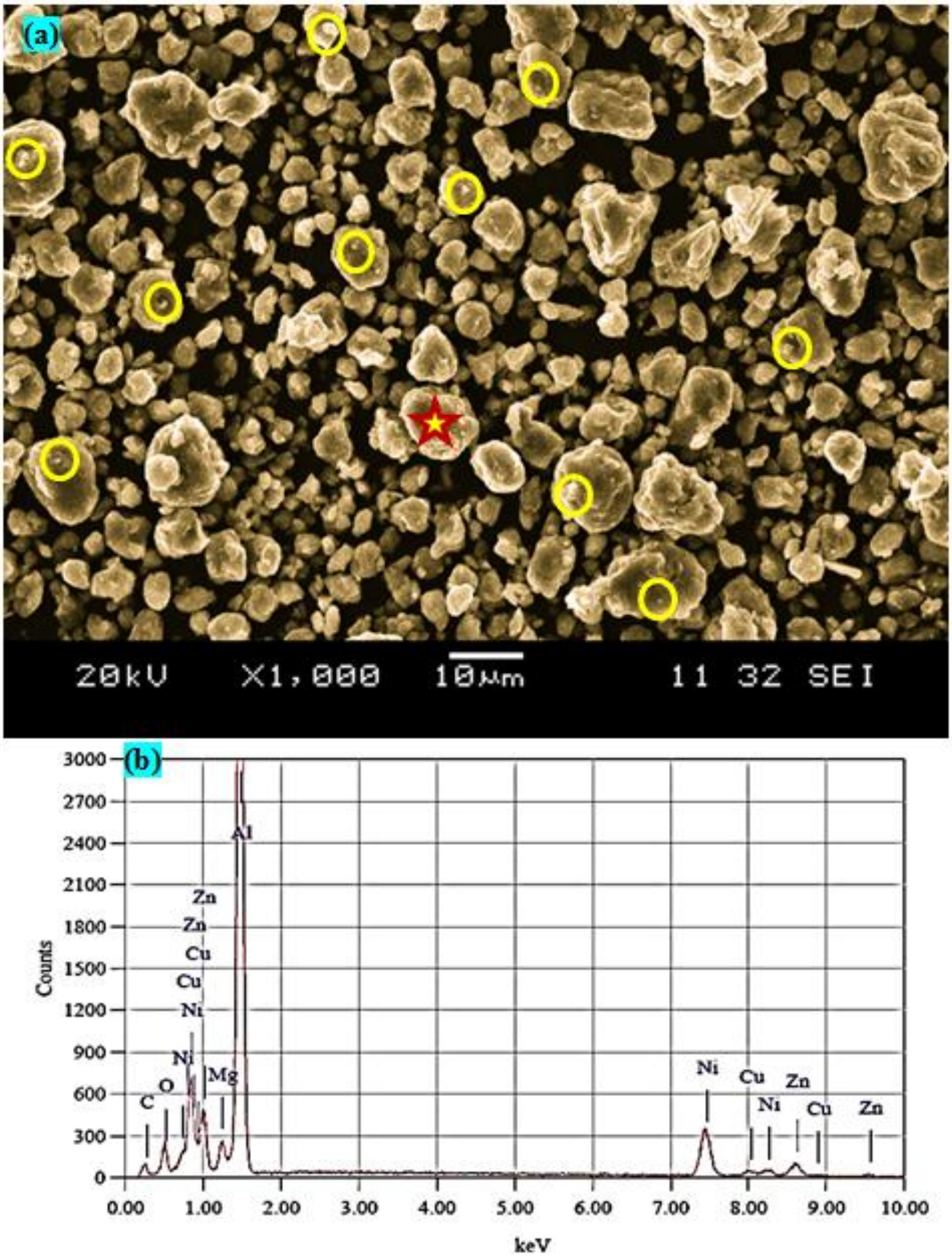

**Figure-3.** The (a) SEM morphology and (b) the EDS scan analysis of the milled powder alloy-**III** at milling times of 15 hrs.

Mechanically alloyed powders show extension of equilibrium solid solubility limits, which has been also achieved for nickel in the milled powders during this study. On mechanically alloying blended elemental powder mixtures, interdiffusion (as shown in Figs. 2 and 3) between the components occurs and solid solutions form. This solid solubility limit is expected to increase with long time of milling as diffusion progresses and reaches a saturation level, beyond which no further extension of solid solubility occurs [19].

The SEM micrograph of specimen of BM alloy-**I** which underwent the sintering process, is shown in Figure 4 (a). The presence of precipitate particles distributed enormously within the Al-matrix (marked by arrows) is evident. The Mapping of EDS scans in Figure 4(b) shows the chemical composition within the labeled region.

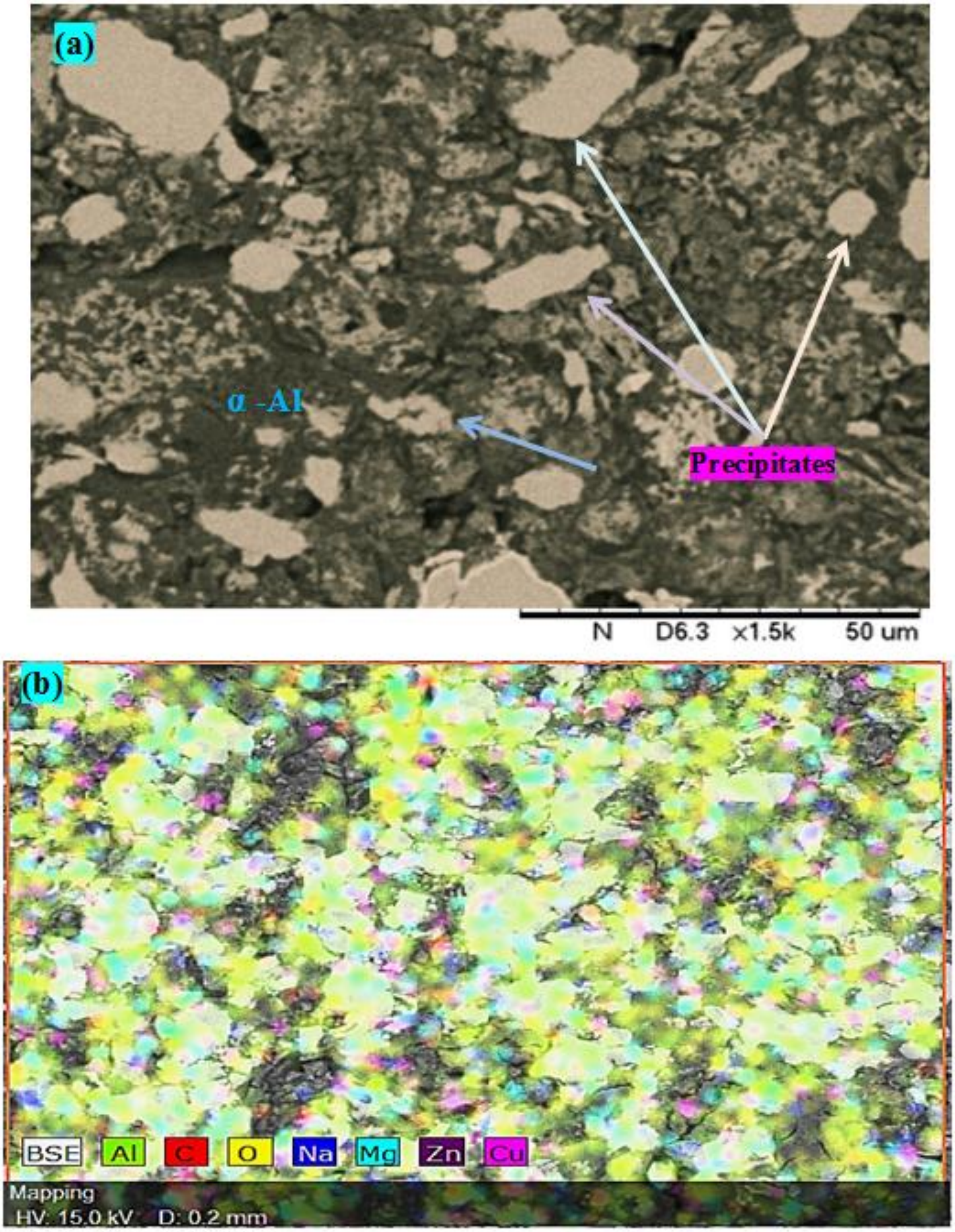

**Figure-4.** The (a) SEM micrograph of the alloy-**I** and (b) EDS scan analysis mapping after performed the sintering process at 600°C/1h.

Shown in Figure 5 (a) is the SEM micrograph of the sintered compact of Alloy-**II** underwent homogenization treatment and then aged at 124 °C. The image shows plenty of intermetallic of Ni-rich compounds distributed and formed within the milled Al-matrix along with precipitates of alloying elements. The EDS mapping microanalysis in Figure 5(b) proved that the formation of immiscible compounds rich in Ni alloyed with Zn, Mg and Cu elements within the Al-matrix was achieved.

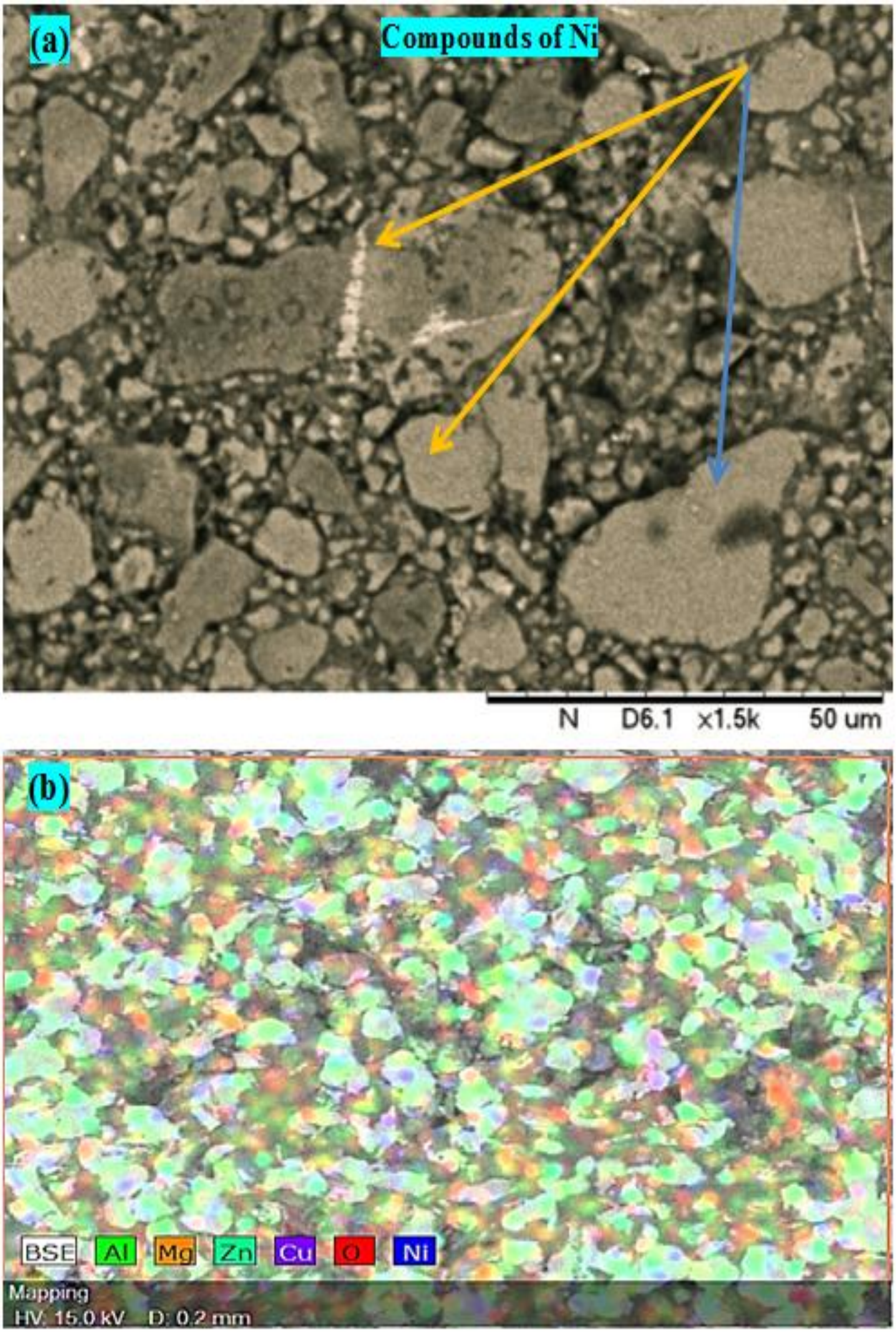

**Figure-5.** The (a) SEM micrograph of the alloy-**II** and (b) EDS scan analysis mapping after applied the aging treatment.

The SEM micrograph of the alloy-**III** compact after the homogenization treatment and the aging is shown in Figure 6. It is clear that numerous dispersion intercompounds of Ni with precipitates embedded within the Al matrix. The depicted EDS scan analysis in Figure 6 (b) reveals the stoichiometry of the defined region in the Al matrix.

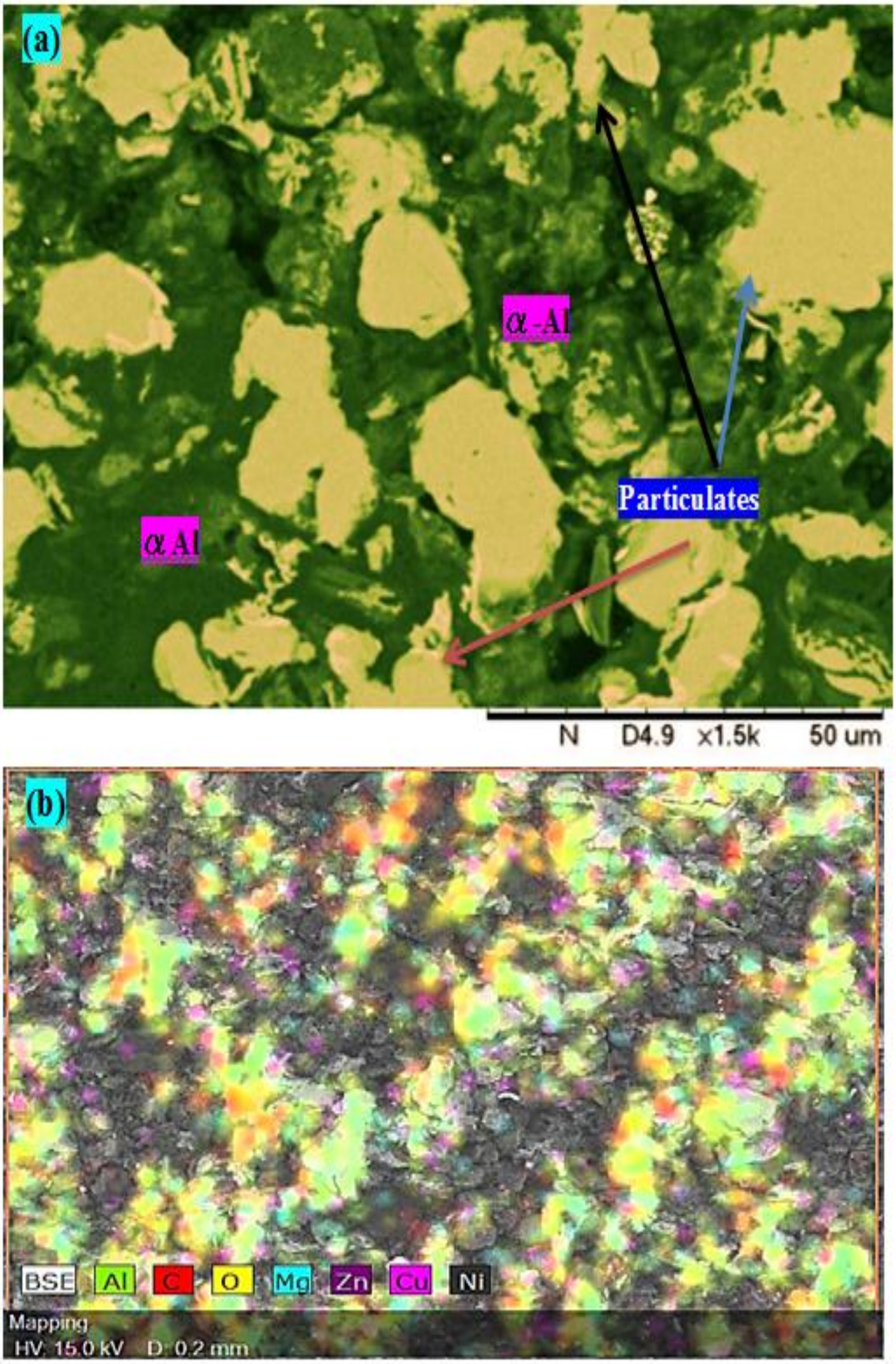

**Figure-6.** (a) SEM micrograph of alloy-**III** and (b) the EDS scan mapping underwent the aging.

Microhardness values of the milled alloyed alloys **I**, **II**, and **III** after the compacting process with the sintering and the aging heat treatments are given in Figure 7. Generally, the improvements attained in hardness for the sintered compacts of different BM alloys after the treatment attributed to the effectiveness of the mechanical alloying brought about by the ball milling action besides roles of the aging.

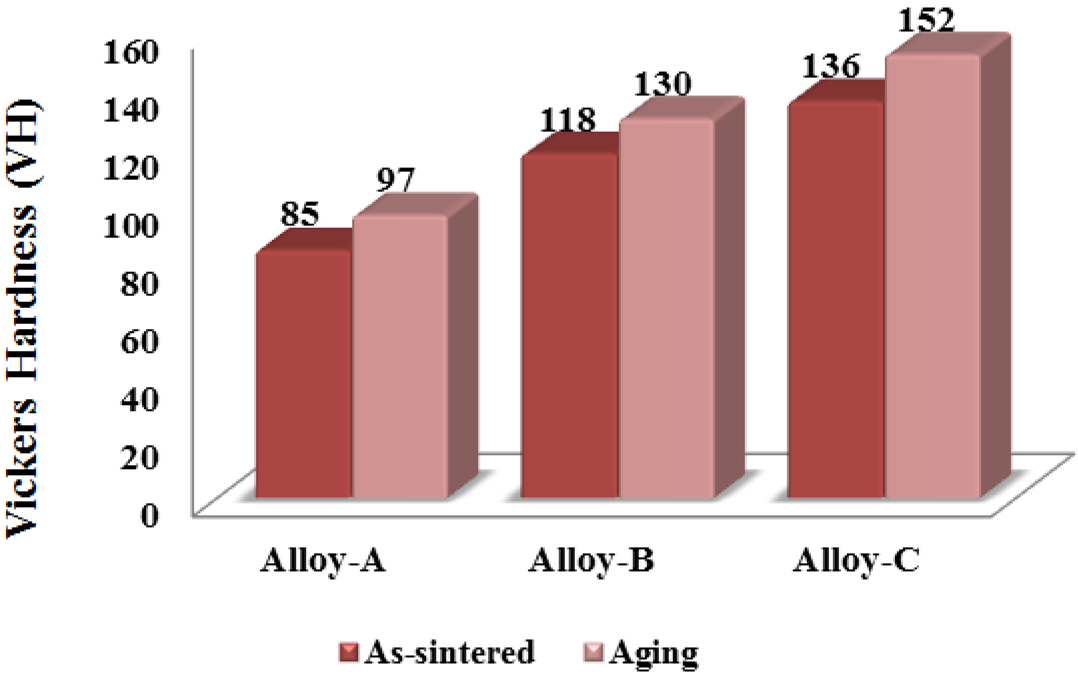

**Figure-7.** Vickers hardness of sintered compacts of the alloys-**I**, **II** and **III** after the aging treatment.

Higher hardness was attained for the aged treated with sintered compacts of alloy **III** (with 16wt. % Ni) as compared to sintered compacts of alloy **I** BM powder that underwent similar treatment. The milled alloys **II** and **III** sintered compacts underwent the aging exhibited higher hardness than what in the milled alloy-**I** underwent the similar conditions and treatments.

Generally, several factors contribute to the hardness and compression strength enhancement of the sintered compacts of alloys **I**, **II** and **III** BM powder;

1. The MA mechanisms via BM process contribute to grain refinement and the formation of the supersaturated solid solution of alloying elements like Zn, Mg and Cu in the Al mixture. Additionally the extension of solubility limits of the Ni in the alloy **II** and **III** BM mixtures is another important reason for strength improvement.
2. Precipitation hardening due to the existence of the compound phases which were formed in the sintered compacts after the application of the aging on the BM alloys.
3. Dispersion strengthening attributed to the presence of intercompounds of Ni-rich within the heat treated sintered compacts of BM **II** and **III**, alloys. The effects of nickel additives dispersed within the Al matrix of alloys. These dispersoid phase particles are looped, by passed and/or sheared by the dislocation through the Orowan mechanism as follow [14].

$$\tau_0 = \frac{G\,b}{\lambda} \qquad (1)$$

where τ0 is the stress required for a dislocation to counter the reinforcement, G is the shear modulus of the material, and b is the Berger's vector of the dislocation. The stress required to move a dislocation around a particle is the yield strength. Thus, the increase in the yield strength means enhancement in mechanical properties.

## 4. CONCLUSIONS

In this study, the high-energy ball milling method was used to synthesize Al-Ni intermetallic compounds within Al-Zn-Mg-Cu mixture powders. This study on the different characteristics of the compound led to the following results:

1. Observations of microstructure indicate that reduction in the grain sizes of milled powders **I**, **II** and **III** was achieved in the milling stages. However, with long milling time, grain size approaches the steady-state condition. Furthermore, diffused and homogeneous nickel atoms are attached to the mixture during MA.
2. The mechanical alloying mechanism allows for the attainment of Al solid solutions with  mounts ofnickel and dopants Zn, Mg, and Cu at a concentration that exceeds their equilibrium solubility compared with melting and crystallization in conventional processes.
3. Vickers hardness values of the milled alloys **I**, **II**, **III** after applying the aging was higher than that of after sintering. However, owing to nickel alloy **III** possessed the highest Vickers hardness.

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