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ATMOSPHERE CONTROLLED INDUCTION SYSTEM**

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Mg₆₅Ni₂₀Y_{15-x}Ag_x (X=1, 2, 3, 5) ALLOYS PREPARED VIA ATMOSPHERE CONTROLLED INDUCTION SYSTEM

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Abstract

In this work, Mg₆₅Ni₂₀Y_{15-x}Ag_x (X= 1, 2, 3, 5) alloys were manufactured by atmosphere controlled induction system. The effect of Ag ratio on the microstructural properties, micro-hardness, density and homogeneity of the Mg-Ni-Y alloys were investigated. These alloys were characterized by X-ray diffraction (XRD), optical microscopy (OM), scanning electron microscopy with energy dispersive X-ray (SEM-EDX) and Vickers microhardness measurement. According to XRD results, Ni₃Y and Mg₆Ni phases were observed as well as AgY and Ag₁₇Mg₅₄ phases which were obtained in alloys. The quantitative results of EDX analysis confirm that the chemical composition of the obtained phases is very close and their homogeneities are so high. The average microhardness values of the ingot alloys were measured between 208 and 266 HV for matrix. The elastic modulus and densities of the Mg₆₅Ni₂₀Y_{15-x}Ag_x (X= 1, 2, 3, 5) alloys increased by increasing Ag into the alloys and they were determined in the range of 58.18 – 68.12 GPa and 3.14 – 3.53 g/cm³, respectively.

Keywords: Mg based alloys, Microhardness, Elastic Modulus, Arc Melting, XRD

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1. Introduction

Magnesium (Mg) is one of the most abundant element in the earth crust [1] and it is mostly used for engineering application with being alloyed with other metals [2]. Mg-based alloys have perfect heat resistivity, vibration absorptivity and good recyclability [3]. Although, they are commonly known from an automobile and aerospace applications [1, 4], there are also different application areas in medicine [5-7], electronic [8], sports and military equipment [9]. Nowadays hydrogen storage properties, the new application area, of Mg-based alloys has been studied extensively since material availability, weight, storage capacity and cost of Mg element is considered to be one of the best suitable material [3, 10, 11].

Mg-Ni-Y alloys were considered to be promising systems to develop new alloys with some functional applications [12]. Long period stacking ordered (LPSO) structure and hydrogenation-dehydrogenation properties of this type of ternary magnesium based alloys were studied extensively in literature [13-16]. Many Mg-Ni-Y alloys with different composition rates such as $\text{Mg}_{91}\text{Ni}_{4.5}\text{Y}_{4.5}$ [17], $\text{Mg}_{90.5}\text{Ni}_{3.25}\text{Y}_{6.25}$ [18], $\text{Mg}_{85}\text{Ni}_6\text{Y}_9$ [19], $\text{Mg}_{88}\text{Ni}_5\text{Y}_7$ [20], $\text{Mg}_{91.9}\text{Ni}_{4.3}\text{Y}_{3.8}$ [21], were reported in terms of LPSO structures. Moreover, thermodynamic interpretation of Mg-Ni-Y based alloys were also characterised by Mezbahul-Islam and Medraj [22]

The quaternary Mg-based alloys especially in the form Mg-Ni-Y-X alloys are needed to be examined, because of the reason that there are very few data in literature on these types of alloys. Mg-Ni-Y-C based alloy produced by two different methods [23] and microstructural properties of this alloys were investigated [24]. Microstructural and electrochemical properties of amorphous Mg-Ni-Y-La alloy was studied by Wenxian et al [25].

The present study deal with producing, $\text{Mg}_{65}\text{Ni}_{20}\text{Y}_{15-x}\text{Ag}_x$ ($X= 1, 2, 3, 5$) alloys by atmosphere controlled induction technique. These alloy systems with at. % 65 Mg, % 20 Ni and % 15 Y have been chosen because they haven't been worked by adding Ag (at. % 1, 2, 3,

5) in literature, yet. Furthermore, the influence of Ag ratio on the microstructural properties, micro-hardness, elastic modules, density and homogeneity of the Mg-Ni-Y alloys were investigated systematically.

2. Experimental

In this study, $\text{Mg}_{65}\text{Ni}_{20}\text{Y}_{15-x}\text{Ag}_x$ ($x=1, 2, 3, 5$) alloys were prepared under atmosphere controlled induction system using pure Mg (% 99.8), Ni (% 99.5), Y (% 99.8) and Ag (% 99.7). Firstly, appropriate amount of metals was placed in an alumina crucible which has a hole at the bottom. Also, the hole was plugged with an alumina stopper. Before heating, the crucible was encapsulated with a glass chamber. In order to control the atmosphere of the chamber, high purity argon gas was continuously flushed. After a proper melting, the liquid metal mixture was poured into a glass tube placed on copper plate. The cast ingots were sliced with a precision diamond saw in order to prepare sample for characterisation. Rigaku Dmax2200 using CuK_α was employed for XRD examinations. One slice of the alloy was cold moulded in epoxy resin to prepare the sample for microstructural examination. The surface of the sample was ground with 80-2400 emery paper and polished with alumina in a suspension. Before SEM investigation, microhardness of the polished samples were measured by forming indents by applying 50 gf using Mitutoyo HM-220 microhardness tester. SEM and SEM-EDX investigations were carried out with a Philips XL30 FEI SEM with an EDX detector. Also, elastic modulus measurements of the alloys based on flexural vibration resonance were made with GrindoSonic Mk5.

3. Results and Discussion

XRD patterns of the $\text{Mg}_{65}\text{Ni}_{20}\text{Y}_{15-x}\text{Ag}_x$ ($x=1, 2, 3, 5$) alloys are presented in Fig. 1. In the XRD patterns shown in Fig. 1a-d, $\text{Mg}_{65}\text{Ni}_{20}\text{Y}_{15-x}\text{Ag}_x$ ($x=1, 2, 3, 5$) alloys have four different phases such as Ni_3Y , Mg_6Ni , $\text{Ag}_{17}\text{Mg}_{54}$ and AgY . Among these phases, Ni_3Y phase

at observed around $2\theta=43^\circ$ shows the strongest diffraction peaks, which clearly evidence that the volume fraction of Ni_3Y phase is the largest. Although the content of Ag in the alloy was rather small, the AgY phase was also observed, this is probably because the solid solubility of Ag in Y is very limited. On the other hand, by increasing the Ag content in the alloys, the peak intensity of the AgY phase increased. As can be seen clearly in Fig. 1a, the crystalline diffraction peaks are few. However, many peaks occur in Fig. 1b and Fig. 1c which belong to $\text{Mg}_{65}\text{Ni}_{20}\text{Y}_{12}\text{Ag}_3$ and $\text{Mg}_{65}\text{Ni}_{20}\text{Y}_{13}\text{Ag}_2$ alloys, respectively. This difference stems from the Ag amounts in the alloys. Furthermore, the more AgY phases are observed in $\text{Mg}_{65}\text{Ni}_{20}\text{Y}_{12}\text{Ag}_3$ (Fig. 1b) and $\text{Mg}_{65}\text{Ni}_{20}\text{Y}_{13}\text{Ag}_2$ (Fig. 1c) alloys than in $\text{Mg}_{65}\text{Ni}_{20}\text{Y}_{14}\text{Ag}_1$ alloy because of the higher Ag rate. It should be mentioned that, no evidences of pure elemental Mg, Y, Ni or Ag peaks were observed. Therefore each constituents which occurred the ingot alloys interacted effectively with one another. As shown in Fig. 1d, $\text{Ag}_{17}\text{Mg}_{54}$ phase was obtained only in $\text{Mg}_{65}\text{Ni}_{20}\text{Y}_{10}\text{Ag}_5$ alloy. It wasn't seen other Mg-based alloys. This may be attributed to Ag (at. % 5) rate in the alloy. Therefore, the increasing of the Ag rate in the Mg-Ni-Y alloy system can be caused to occur $\text{Ag}_{17}\text{Mg}_{54}$ phase.

Fig. 2 shows the SEM micrographs of the $\text{Mg}_{65}\text{Ni}_{20}\text{Y}_{15-x}\text{Ag}_x$ ($x=1, 2, 3, 5$) alloys with different compositions. The microstructure of the alloys consists of plate-like (light grey), irregular shaped precipitates and lamellar structures (dark grey stripes) (Fig. 2a-d). It seems the thickness of plate-like precipitates decreases though the length of them increases with Ag amount (Fig. 2a-b) and the precipitates are rather in needle-like morphology for the alloy with 3 at.% Ag (Fig. 2c). Furthermore these precipitates almost disappear with 5 at.% Ag in the alloys (Fig. 2d). As can be seen clearly from Fig. 2a-d, the surface morphology of $\text{Mg}_{65}\text{Ni}_{20}\text{Y}_{14}\text{Ag}_1$, $\text{Mg}_{65}\text{Ni}_{20}\text{Y}_{13}\text{Ag}_2$ and $\text{Mg}_{65}\text{Ni}_{20}\text{Y}_{12}\text{Ag}_3$ alloys have similar properties while $\text{Mg}_{65}\text{Ni}_{20}\text{Y}_{10}\text{Ag}_5$ (Fig. 2d) alloy has relatively different surface shapes. This is because Ag ratio into the $\text{Mg}_{65}\text{Ni}_{20}\text{Y}_{10}\text{Ag}_5$ alloy is more than the other alloys. Besides, AgY phase are observed into $\text{Mg}_{65}\text{Ni}_{20}\text{Y}_{14}\text{Ag}_1$, $\text{Mg}_{65}\text{Ni}_{20}\text{Y}_{13}\text{Ag}_2$ and $\text{Mg}_{65}\text{Ni}_{20}\text{Y}_{12}\text{Ag}_3$ alloys. However,

$\text{Ag}_{17}\text{Mg}_{54}$ phase is only obtained into the $\text{Mg}_{65}\text{Ni}_{20}\text{Y}_{10}\text{Ag}_5$ alloy. This difference changes the surface morphology of the $\text{Mg}_{65}\text{Ni}_{20}\text{Y}_{10}\text{Ag}_5$ alloy.

Furthermore, Fig. 3a shows the higher magnification SEM micrograph of this alloy ($\text{Mg}_{65}\text{Ni}_{20}\text{Y}_{10}\text{Ag}_5$) with marked spots where SEM-EDX analysis was carried out. The typical SEM-EDX spectrums obtained from these certain spots of 1, 2 and 3 are given in Fig. 3b, 3c and 3d, respectively with their semi-quantitative elemental compositions. The irregular shaped precipitates (Spot 1) present in SEM images of all samples, are rich in Mg, Y and especially Ni. The dark grey stripes (Spot 3) are rich in Mg and Ag though their surrounding areas (Spot 2) are rich in Mg but poor in Ag. Light grey plate/needle-like precipitates are rich in Ni and the matrix surrounding the light grey plate/needle-like precipitates are rich in Mg and includes almost 10 at.% Ni and Y though the corresponding SEM-EDX spectrums are not given.

Fig. 4 shows the typical OM micrographs of $\text{Mg}_{65}\text{Ni}_{20}\text{Y}_{15-x}\text{Ag}_x$ ($X=1, 2, 3, 5$) alloys. As seen clearly from Fig. 4a-d, the phases, Ni_3Y , Ni_7Y_2 , Mg_6Ni and AgY , which are marked by XRD data are shown by different shapes. These shapes have similar appearance with SEM micrographs. It is interesting that the phase shapes change from Fig. 4a to Fig. 4d. although the same phases exist in the ingot alloy systems. It is due to increasing of Ag rates into the Mg-Ni-Y. Moreover, it might also cause cooling rate of the melt alloys.

The mechanical properties and densities of the $\text{Mg}_{65}\text{Ni}_{20}\text{Y}_{15-x}\text{Ag}_x$ ($X=1, 2, 3, 5$) alloys are listed in Table 1 and Table 2. As listed in Table 1, the microhardness values of the ingot alloys relatively increase by increasing Ag rates. The microhardness changes of the alloys were determined in two ways which are precipitation and matrix. According to the obtained results, precipitation microhardness of the $\text{Mg}_{65}\text{Ni}_{20}\text{Y}_{15-x}\text{Ag}_x$ ($X=1, 2, 3, 5$) alloys were changed in the range of 254-306 HV. The measured microhardness values of the alloys in the matrix increased as long as Ag quantity increase into the Mg-Ni-Y and they were determined between 208 HV and 256 HV. The elastic modulus of the $\text{Mg}_{65}\text{Ni}_{20}\text{Y}_{15-x}\text{Ag}_x$ ($X=1, 2, 3, 5$)

alloys are listed in Table 2. As can be understood clearly, the elastic modulus of the alloys increased with increasing Ag rates into the Mg-Ni-Y. The elastic modulus of $\text{Mg}_{65}\text{Ni}_{20}\text{Y}_{15-x}\text{Ag}_x$ ($X=1, 2, 3, 5$) alloys were measured 58.18, 61.94, 65.91 and 66.62 GPa, respectively. Therefore, it can be concluded that the increasing Ag quantity into the ingot alloys effect on positively the mechanical properties of $\text{Mg}_{65}\text{Ni}_{20}\text{Y}_{15-x}\text{Ag}_x$ ($X=1, 2, 3, 5$) alloys.

Table 2 gives also densities of the $\text{Mg}_{65}\text{Ni}_{20}\text{Y}_{15-x}\text{Ag}_x$ ($X=1, 2, 3, 5$) alloys. The density of the ingot alloys increase by increasing Ag quantity into the alloy systems. Thus, the densities of the $\text{Mg}_{65}\text{Ni}_{20}\text{Y}_{15-x}\text{Ag}_x$ ($X=1, 2, 3, 5$) were determined 3.14, 3.24, 3.37 and 3.53 g/cm^3 , respectively. These results can be explained with density of Ag which has the highest value in the ingot alloy systems. According to this, as the amount of Ag increased, the density of the $\text{Mg}_{65}\text{Ni}_{20}\text{Y}_{15-x}\text{Ag}_x$ ($X=1, 2, 3, 5$) ingot alloys increased directly proportional.

4. Conclusions

In the present study, the $\text{Mg}_{65}\text{Ni}_{20}\text{Y}_{15-x}\text{Ag}_x$ ($X=1, 2, 3, 5$) alloys were successfully synthesized by atmosphere controlled induction system. The following conclusions are drawn:

1. The phases such as Ni_3Y and Mg_6Ni were obtained as well as AgY and $\text{Ag}_{17}\text{Mg}_{54}$ phases in the ingot alloys after production processes.
2. The identified phases above were observed SEM and OM images with different shapes and these phases were also confirmed by EDX results.
3. The average microhardness values of $\text{Mg}_{65}\text{Ni}_{20}\text{Y}_{15-x}\text{Ag}_x$ ($X=1, 2, 3, 5$) alloys were measured between 208 and 301 HV. It was determined that the effect of Ag ratio on microhardness of the ingot alloys was positive.
4. The elastic modulus values increased by increasing Ag rates into the alloys and they were calculated 58.18, 61.94, 65.91 and 66.62 GPa for $\text{Mg}_{65}\text{Ni}_{20}\text{Y}_{15-x}\text{Ag}_x$ ($X=1, 2, 3, 5$) alloys, respectively.

5. The density of $\text{Mg}_{65}\text{Ni}_{20}\text{Y}_{15-x}\text{Ag}_x$ ($X=1, 2, 3, 5$) alloys increased because of the increasing Ag into the Mg-Ni-Y systems and they were measured 3.14, 3.24, 3.37 and 3.53 g/cm^3 , respectively.

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For Review Only

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Figures Captions

Figure 1: XRD patterns of as cast alloys, a) $\text{Mg}_{65}\text{Ni}_{20}\text{Y}_{14}\text{Ag}_1$; b) $\text{Mg}_{65}\text{Ni}_{20}\text{Y}_{13}\text{Ag}_2$; c) $\text{Mg}_{65}\text{Ni}_{20}\text{Y}_{12}\text{Ag}_3$; d) $\text{Mg}_{65}\text{Ni}_{20}\text{Y}_{10}\text{Ag}_5$

Figure 2: SEM images of as-cast a) $\text{Mg}_{65}\text{Ni}_{20}\text{Y}_{14}\text{Ag}_1$; b) $\text{Mg}_{65}\text{Ni}_{20}\text{Y}_{13}\text{Ag}_2$; c) $\text{Mg}_{65}\text{Ni}_{20}\text{Y}_{12}\text{Ag}_3$; d) $\text{Mg}_{65}\text{Ni}_{20}\text{Y}_{10}\text{Ag}_5$

Figure 3: SEM image and EDX results from specific spots of as-cast $\text{Mg}_{65}\text{Ni}_{20}\text{Y}_{10}\text{Ag}_5$ alloys
a) SEM image; b) Spot 1; c) Spot 2; d) Spot 3.

Figure 4: Optical micrographs of as cast a) $\text{Mg}_{65}\text{Ni}_{20}\text{Y}_{14}\text{Ag}_1$; b) $\text{Mg}_{65}\text{Ni}_{20}\text{Y}_{13}\text{Ag}_2$; c) $\text{Mg}_{65}\text{Ni}_{20}\text{Y}_{12}\text{Ag}_3$; d) $\text{Mg}_{65}\text{Ni}_{20}\text{Y}_{10}\text{Ag}_5$

Table Captions

Table 1: Microhardness values of $\text{Mg}_{65}\text{Ni}_{20}\text{Y}_{15-x}\text{Ag}_x$ ($x=1, 2, 3, 5$) alloys

Alloys	Precipitation Microhardness (HV)	Matrix Microhardness (HV)
$\text{Mg}_{65}\text{Ni}_{20}\text{Y}_{14}\text{Ag}_1$	301	212
$\text{Mg}_{65}\text{Ni}_{20}\text{Y}_{13}\text{Ag}_2$	275	208
$\text{Mg}_{65}\text{Ni}_{20}\text{Y}_{12}\text{Ag}_3$	254	266
$\text{Mg}_{65}\text{Ni}_{20}\text{Y}_{10}\text{Ag}_5$	306	256

Table 2: Elastic modulus and densities of $\text{Mg}_{65}\text{Ni}_{20}\text{Y}_{15-x}\text{Ag}_x$ ($x=1, 2, 3, 5$) alloys

Alloys	Elastic Modulus (GPa)	Density (g/cm^3)
$\text{Mg}_{65}\text{Ni}_{20}\text{Y}_{14}\text{Ag}_1$	58.18	3.14
$\text{Mg}_{65}\text{Ni}_{20}\text{Y}_{13}\text{Ag}_2$	61.94	3.24
$\text{Mg}_{65}\text{Ni}_{20}\text{Y}_{12}\text{Ag}_3$	65.91	3.37
$\text{Mg}_{65}\text{Ni}_{20}\text{Y}_{10}\text{Ag}_5$	68.62	3.53

Figure 1A

Figure 1B

Figure 1C

Figure 1D

Figure 2

Figure 3A – SEM for EDX

Figure 3B, C, D - EDX

Figure 4