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Physical properties of the multifunctional $\text{Mg}_{80}\text{Ni}_{10}\text{Gd}_{10}$ alloy

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Abstract:

The focus of this work is on exploring the effect of cooling rate on the microstructure and hardness of the $\text{Mg}_{80}\text{Ni}_{10}\text{Gd}_{10}$ alloy which is known for its potential hydrogen storage capability. A series of chart is displayed to compare the mechanical and transport properties with commercial Mg-based alloys, Mg-based metallic glasses and light high entropy alloys. We outline the interest of this alloy composition for the development of new light high entropy alloy with multifunctionality.

Strategic areas such as transport and energy put a great challenge on material design because these applications require a combination of structural and functional properties. For example, in the automotive industry the primary eco-objective is to reduce the fuel consumption and the CO₂ emissions which depend on vehicle weight and propulsion system. To this respect, magnesium alloys are of great interest because they can address both of these issues.

As structural materials, they represent the lightest currently used class of metallic alloys and offer a mean to save weight by replacing heavier steel and Al alloy components. The mechanical properties of these alloys are mainly controlled by the nature of the interactions between dislocations and the microstructure. The key concept is dislocation pinning, and the spacing and effectiveness of the obstacles introduced as pinning points: there are solute atoms, secondary phase precipitates and other dislocations. For some alloys, referred to as precipitation hardened, the total hardening response is dominated by the contribution arising from the precipitates [1] and can be enhanced by trace additions of selected elements leading to a refinement in the particle distribution [2]. When applications require improved elevated temperature strength and creep resistance, rare earth can be added such as Ce, La and Nd [3] in the EQ, EZ, QE, WE, and ZE series. Besides, Mg can form bulk metallic glasses in the Mg-TM-RE (TM: transition metal, RE: rare earth) [4] and Mg-Ca-Zn systems [5] with unique properties making these alloys attractive for use in biomedical applications. As a last example, Mg can be added to Fe to produce a low-density steel with improved specific properties [6].

As functional materials, Mg alloys are materials of choice because of their potential high hydrogen storage capability. It can reach 7.6 wt.% in the case of pure magnesium with formation of MgH₂, but its use is limited by the high stability of the hydride and slow sorption kinetics. Tuning the stability of the hydride can be achieved by alloying in order to replace Mg-Mg bonds by Mg-TM (mostly unstable hydrides) or Mg-RE bonds (stable hydrides). Several

studies have been reported on the hydrogenation properties of Mg-TM [7] and Mg-TM-RE alloys [8, 9], in particular the ternary system Mg-Ni-Gd.

Recently, a new branch of the metallic alloy tree, called high entropy alloys (HEAs), receives considerable attention because it changes the paradigm of traditional alloy design strategy [10]. HEAs have no base element in contrast with conventional metallic alloys constituted of one major element and several minor additions of alloying elements. The main idea behind the development of HEAs is to promote the formation of entropy-stabilized single-phase solid solution. This new strategy opens up new opportunities for the discovery of new alloys with better properties through the exploration of the vast unknown interior region of the composition space. There are a limited number of studies on low density HEAs based on elements such as Al, Be, Li, Mg, Sc, Si, Sn, Ti and Zn [11-13]. Despite their potential interest for weight saving, these alloys suffer from several drawbacks: they are brittle and difficult to process due to the broad range of melting and boiling points, and high oxygen affinity of the elements used, so that mechanical alloying is usually applied. Similarly only few studies have been reported on functional properties of HEAs. They concern mainly the electrical [14, 15], thermal [14, 16] and magnetic [16, 17] properties of 3d transition metal HEAs.

In this paper we explore the microstructure, mechanical and transport properties of the $\text{Mg}_{80}\text{Ni}_{10}\text{Gd}_{10}$ alloy with emphasis on the effect on the cooling rate during solidification. This alloy was selected because it represents an interesting candidate in the search of multifunctionalities: it exhibits a good hydrogenation capability for energy storage [18] and it can be considered as a base for new light structural HEAs, *i.e.* $\text{Mg}_{80}(\text{Co,Cu,Fe,Ni})_{10}(\text{Ce, La, Nd,Y})_{10}$. Whereas Mg crystallizes in an hcp lattice, it has been shown that alloys with composition ranging from $\text{Mg}_{72-78}\text{Ni}_{8-12}\text{Gd}_{10-15}$ are single-phase crystallizing in a cubic structure [18]. Additionally, single-phase were also observed for compositions $\text{Mg}_{80}\text{Cu}_{10}\text{RE}_{10}$ (with RE = La, Tb, Y) [19, 20].

Samples with composition $\text{Mg}_{80}\text{Ni}_{10}\text{Gd}_{10}$ were prepared by induction melting of gadolinium pieces (Stream Chemicals, > 99.9 %), nickel rod (Stream Chemicals, > 99.9 %), and magnesium rod (alpha Aesar, > 99.8 %) in Ta tubes sealed under an argon pressure of 800 mbar to prevent evaporation and oxidation. In addition, the surfaces of both magnesium and gadolinium pieces were cleaned in a glove box and the argon filler was purified on a magnesium sponge bed heated to 673K. After solidification, two ingots thus produced were annealed at 1373 K in their Ta tube placed in a quartz ampoule sealed under vacuum and then cooled to room temperature at different cooling rates: 2K/s for the furnace cooled sample (FC), and of about 100K/s for the air cooled sample (AC). A third ingot was casted into a water-cooled copper mold (3 mm in diameter) at a cooling rate of about 10^4 K/s (sample CC). A fourth ingot was melt spun onto a copper wheel spinning at a speed of 30 m/s to form rapidly-solidified ribbons (cooling rate of about 10^6 K/s) a few tens of micrometers in thickness (sample named MS). Melt spinning and copper mold casting were done using melt spinner SC from Edmund Bulhler GmbH. The experiments were performed under purified argon. No oxidation was observed.

The four samples were characterized by X-ray powder diffraction using a Philips PW 1050 diffractometer with $\text{CuK}\alpha$ radiation ($\lambda K_{\alpha 1} = 1.5406 \text{ \AA}$, $\lambda K_{\alpha 2} = 1.5443 \text{ \AA}$). These patterns were scanned by steps of 0.02° (2θ) from 5° to 80° with a constant counting time of 30 s. The crystallite sizes were evaluated using Scherrer formula.

Metallographic examination was performed by scanning electron microscopy (SEM) in Back Scattered Electron (BSE) on a Tescan VEGA II-SBH working at 10 and 20 kV with a current beam of 90 μA . The samples were polished with different silica and diamond pastes without water. Phase's composition was investigated by electron probe microanalyses (EPMA, CAMECA SX100) with metallic Gd (L_α), Ni (K_α) and Mg (K_α) as standards.

Nanoindentation tests were carried out using a NT 600 nanoindenter manufactured by Micro Materials Limited (Wrexham, Great Britain) having a load resolution of 0.1 nN, a displacement

resolution of 0.01 nm, and an Berkovitch type indenter (triangular pyramid). Numerous nanoindentation measurements were performed for each tested samples applying a loading rate of 5 mN/s and a maximum load of 500 mN. The reduced modulus E_R and the hardness H were extracted from the penetration load–displacement curves of the indenter using the Oliver and Pharr analysis [21] using the expression:

$$1/E_r = (1-\nu^2)/E + (1-\nu_i^2)/E_i$$

where E and ν are Young's modulus and Poisson's ratio for the tested specimen, E_i and ν_i are Young's modulus and Poisson's ratio for the indenter (diamond), respectively 1141 GPa and 0.07.

$$H = P_{\max}/A$$

where $P_{\max}$ is the maximum load and A is the measured projected area of the hardness impression.

The thermal conductivity was determined as a function of the temperature according to the relationship $\kappa = \alpha d C_p$, where α is the thermal diffusivity measured with the flash diffusivity method (LFA 457 MicroFlash), d the density and C_p the heat capacity measured with the differential scanning calorimetry technique (PerkinElmer DSC8500). The electrical resistivity, ρ , was measured as a function of the temperature using the four-probe method.

The X-ray diffraction patterns of the $Mg_{80}Ni_{10}Gd_{10}$ alloy for various cooling rates are shown in **Figure 1**. For samples FC, AC and CC the main peaks (indicated by a circle) correspond to an average face-centered cubic structure (fcc) with $a \approx 4.55 \text{ \AA}$. The most intense peak at $2\theta \approx 34^\circ$ corresponds to the (111) Bragg peak. As previously described by Couillaud et al. [18] for a similar composition ($Mg_{78}Ni_9Gd_{13}$), a modulated structure is observed when the crystallinity is high. Then, the satellite (extra) peaks can be indexed by considering vectors of modulation. When the cooling rate is increasing, the signal-to-noise ratio of the diffractograms is decreasing,

and the width of the peaks increase until getting a broad peak for the melt-spun sample. Both signal-to-noise and width variations are a proof of the progressive atomic disorganization of the sample with increasing cooling rate, leading to the amorphization of the melt-spun alloy. The amorphous nature of the MS sample is confirmed by the DSC measurement at a heating rate of 20 K/min shown in **Figure 2**. The thermal behaviour of the MS sample is typical to the amorphous alloys and is characterized by an endothermic reaction due to the glass transition, followed by exothermic reactions corresponding to crystallizations and, at higher temperatures, endothermic reactions corresponding to the melting temperature. These results are in good agreement with the results of Park et al. [22] which highlight a critical diameter of 2 mm for the amorphization of a composition close to $\text{Gd}_{13}\text{Ni}_9\text{Mg}_{78}$. The measured values of the characteristic glass transition, crystallization, and melting temperatures are 187, 207, and 544°C, respectively. $\text{Mg}_{80}\text{Ni}_{10}\text{Gd}_{10}$ amorphous alloy has a supercooled liquid region, $\Delta T_x = 30^\circ\text{C}$) and a glass forming ability indicator, $\gamma = T_x/(T_g+T_m)$, of 0.376. The present alloys exhibit similar T_g , T_x and glass stability values compared to previously reported Mg-based glasses [23, 24].

The evolution of the microstructure of the $\text{Mg}_{80}\text{Ni}_{10}\text{Gd}_{10}$ alloy as a function of the cooling rate is shown on the SEM-BSE images of **Figure 3**. Sample FC presents a relatively homogenous microstructure and the EPMA analysis does not reveal any significant difference on the composition of the two slight contrasts. The average composition is $\text{Mg}_{81}\text{Ni}_8\text{Gd}_{11}$ very close to the nominal alloy composition. In spite of a close diffraction pattern than the one of sample FC (**Figure 1**), sample AC consists of two phases. In BSE imaging mode, the darkest phase in the images has the lowest atomic number and by EPMA has the composition $\text{Mg}_{85}\text{Ni}_5\text{Gd}_{10}$, and the bright phase has the composition $\text{Mg}_{77}\text{Ni}_8\text{Gd}_{15}$. The sample CC is composed of bright grains with composition $\text{Mg}_{78}\text{Ni}_{10}\text{Gd}_{12}$ surrounded by a dark matrix with composition $\text{Mg}_{83}\text{Ni}_7\text{Gd}_{10}$.

The size of the bright grains vary across the section of the ingot from fine grains ($< 5 \mu\text{m}$ in diameter) at the edge in contact with the copper mold to coarse grains ($10\text{-}20 \mu\text{m}$ in diameter) at the center, which can be attributed to the gradient of cooling rate. The sample MS is a single-phase with composition $\text{Mg}_{79}\text{Ni}_9\text{Gd}_{12}$.

SEM and XRD analysis evidence the influence of the cooling rate on the microstructure and crystallinity of the $\text{Mg}_{80}\text{Ni}_{10}\text{Gd}_{10}$ as-solidified alloy:

- At low cooling rate, the alloy consists of a single fcc phase having the nominal alloy composition.
- An increase of the cooling rate induces a refinement of the microstructure, a decrease of the crystallinity and favour partitioning. AC and FC alloys are then composed of two phases with the same average fcc crystallographic structure with slight different compositions (Mg-richer and Mg-poorer grains) and sharp phase boundaries.
- Very high cooling rate leads to the amorphization of the alloy.

Table 1 provides the room temperature hardness and Young's modulus of the $\text{Mg}_{80}\text{Ni}_{10}\text{Gd}_{10}$ alloys measured from the nano-indentation tests as a function of the cooling rate. There is a slight decrease of hardness and no significant change of Young's modulus with the cooling rate for crystallized samples (average values of about 150 Hv and 42 GPa, respectively). In contrast, the amorphous alloy (sample MS) presents a higher hardness (197 Hv) and a lower Young's modulus (36 GPa).

In the crystallized two-phase alloys, the Mg-poorer phase exhibits a higher hardness (212 and 167 Hv in AC and CC samples, respectively) and Young's modulus (53 and 51 GPa) than the Mg-rich phase (97 and 107 Hv, and 32 and 33 GPa, respectively). For the crystallized single-

phase alloy, sample FC, the hardness and Young's modulus values are 160 Hv and 44 GPa, respectively, averaging the lowest and highest values obtained for the two-phase alloys.

To compare the properties of the $\text{Mg}_{80}\text{Ni}_{10}\text{Gd}_{10}$ alloys and illustrate how they compete with commercially available Mg-based alloys, Mg-base metallic glasses and light HEAs, two type of materials property spaces are represented in **Figures 4-5** using logarithmic and semi-logarithmic scales, respectively. In this series of charts, individual alloys, shown as circles, are enclosed in large bubbles that represent alloy families, and each family occupies a particular area of space depending on their combination of properties.

Hardness is plotted as a function of the density in **Figure 4**. The dashed line represents the ratio of the hardness over the density, it is a guideline for lighter structure to be made since the hardness is related to the yield strength. Materials above this selection line perform better than materials below. With a much higher hardness and a slight higher density, $\text{Mg}_{80}\text{Ni}_{10}\text{Gd}_{10}$ alloys compete well with commercial Mg-based alloys. Their properties are similar to Mg-based metallic glasses without having constraints of high cooling rates and limitations in achievable maximal sizes. Despite a higher density, the $\text{Al}_{20}\text{Li}_{20}\text{Mg}_{10}\text{Sc}_{20}\text{Ti}_{30}$ HEA is better than $\text{Mg}_{80}\text{Ni}_{10}\text{Gd}_{10}$ alloys. However, light HEAs are known to be brittle which is not the case for AC and FC $\text{Mg}_{80}\text{Ni}_{10}\text{Gd}_{10}$ alloys due to a mixture of hard and soft grains as shown in the inset of **Figure 3** where crack propagation is stopped in the soft phase (dark regions).

Comparison of the Thermal conductivity – Electrical resistivity is shown in **Figure 5**. In this materials property space, $\text{Mg}_{80}\text{Ni}_{10}\text{Gd}_{10}$ has equivalent thermal conductivity to HEAs and pure Gd, and lower than Mg-based commercial alloys, pure Ni and pure Mg. Its electrical resistivity is higher than all the metals and metallic alloys and does not follow the Wiedemann-Franz law which indicates that the heat conduction is dominated by the lattice contribution.

In summary, this work reports the microstructures and the functional and structural properties of the $\text{Mg}_{80}\text{Ni}_{10}\text{Gd}_{10}$ alloys as a function of the thermal history. After high temperature heat treatment, low cooling rate leads to the formation of a single fcc phase whereas, at the other end of the spectrum, very high cooling rate favor the formation of an amorphous phase. Between these two extremums, $\text{Mg}_{80}\text{Ni}_{10}\text{Gd}_{10}$ alloys consist of two phases with the same poorly crystallized average fcc structure and slight different compositions. Such intermetallic phases usually have several sub-lattices than can be occupied by more than one element. For example, the known existence of ternary phases with similar compositions (*e.g.* $\text{Mg}_{80}\text{Cu}_{10}\text{La}_{10}$, $\text{Mg}_{80}\text{Cu}_{10}\text{Tb}_{10}$ and $\text{Mg}_{80}\text{Cu}_{10}\text{Y}_{10}$ [19, 20]), and the extended ternary solubility of the intermetallic phases in the Mg-Cu-Ni system [25] suggest that alloys with formula $[\text{Mg}]_{80}[\text{TM}]_{10}[\text{RE}]_{10}$ may have a random distribution of TM, such as Co, Cu, Fe and Ni, on the second sub-lattice, and RE, such as La, Gd, Nd and Y, on the third sub-lattice. This probable sub-lattice disorder can increase the configurational entropy, enhancing the stability of the single phase high entropy alloy and suggesting that the $\text{Mg}_{80}\text{Ni}_{10}\text{Gd}_{10}$ alloy represents an interesting composition scheme for new light HEA developments.

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Table 1 : Hardness and Young's modulus of the phases observed in the Mg₈₀Ni₁₀Gd₁₀ alloys for different cooling rates.

Sample	Nb of phases	Phase contrast	Phase composition	Nano-indentation	
				Nano-hardness (Hv)	Young modulus (GPa)
Furnace cooled (FC)	1		Mg ₈₁ Ni ₈ Gd ₁₁	160±14	44±2
Air cooled (AC)	2	Bright	Mg ₇₇ Ni ₈ Gd ₁₅	212±12	53±3
		Dark	Mg ₈₅ Ni ₅ Gd ₁₀	97±40	32±6
Copper casted (CC)	2	Bright	Mg ₇₈ Ni ₁₀ Gd ₁₂	167±56	51±4
		Dark	Mg ₈₃ Ni ₇ Gd ₁₀	107±30	33±7
Melt-spun (MS)	1		Mg ₇₉ Ni ₉ Gd ₁₀	197±20	36±9

Figure 1: XRD of the $\text{Mg}_{80}\text{Ni}_{10}\text{Gd}_{10}$ alloy (a) furnace cooled (FC sample), (b) air cooled (AC sample), (c) casted in a water-cooled in copper mold (CC sample) and (d) melt-spun (MS sample).

Figure 2: DSC curves of the $\text{Mg}_{80}\text{Ni}_{10}\text{Gd}_{10}$ melt-spun sample (MS).

Figure 3: SEM-BSE images of the $\text{Mg}_{80}\text{Ni}_{10}\text{Gd}_{10}$ alloy (a) furnace cooled (FC sample), (b) air cooled (AC sample), (c) casted in a water-cooled in copper mold (CC sample) and (d) melt-spun (MS sample). The bright and dark phases in the AC and CC samples are Mg-poorer and Mg-richer grains, respectively.

Figure 4: Materials property space for Hardness vs density of Mg₈₀Ni₁₀Gd₁₀ alloys, conventional Mg-based alloys and light HEA. The dashed line gives performance index light weight structure. This chart was made with the CES EduPack database, Level 3 Aerospace edition [26]. It displays data for about 50 Mg commercial engineering alloys (cast: AE, AJ, AM, AS, AZ, EQ, EZ, QE, WE, ZC, and ZE series; wrought: AZ, EA, WE, Z, ZC, ZK, ZM, and ZW series), 5 Mg-based metallic glasses: Mg₇₀Ca₁₀Al₂₀, Mg₇₄Al₈Ga₅Zn₃, Mg₇₆Al₉Ca₅, Mg₈₀Y₅Ni_{7.5}, Mg₈₅Y₁₀Cu₅, and Mg₉₀Ca_{2.5}Ni_{7.5}, and one light HEA.

Figure 5: Materials property space for Thermal conductivity vs Electrical resistivity. The line gives the Wiedemann-Franz law. This chart was made with the CES EduPack database, Level 3 Aerospace edition [26]. It displays data for about 1800 metallic commercial alloys, in addition to one 3d transition metal HEA. κ_l and κ_e stand for the lattice and electrical thermal conductivity, respectively.