ACTA METALLURGICA SLOVACA
2022, VOL. 28, NO. 2, 85-90

RESEARCH PAPER

PROPERTIES OF Al-Zn-Mg-Cu ALLOY WHEN MODIFIED BY La, Ce, AND THERMAL-MECHANICAL

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Received: 19.02.2022
Accepted: 26.04.2022

ABSTRACT

The influence of La, Ce elements and thermal-mechanical treatment on microstructure and mechanical properties of Al-Zn-Mg-Cu alloy are presented in this study. According to the results, when the alloy was added to the La, Ce elements, after casting, the grain size of samples was around 40-50µm compared to that of without about 65µm; and then these impurity samples attained 30µm after homogeneous mixture the grain sizes. After the cold deformation process, the distance between plates is 10µm. This homogenization process contributes to increasing the ductility of the studied alloy. In addition, the EDS lines study shows that after the combination of the deformation and heat treatment, the information of elements mainly focuses on the boundary and in the grain. After recrystallization annealing, the grain size is around 10 µm with the modification sample. Further, as a result of ability deformation from the tensile test, these results demonstrate that the tensile test obtained 140 % when adding La, Ce contents into the alloy combined with thermal-mechanical treatment. The combined uses of La; Ce and thermal-mechanical treatment have increased the ductility of Al-Zn-Mg-Cu alloy. The combination of modification and thermal-mechanical treatment has created a small grain size for the studied alloy.

Keywords: modification; thermal-mechanical, deformation, homogeneous, recrystallization annealing

INTRODUCTION

Superplastic materials were produced as a huge success in the mechanical industry. There are many certainly prominent advantages compared to conventional metals such as the ability to fabricate the thinner parts with the thick more uniformity, the shape more complicated, to save material source, reduction of mass, and increasing the effective use of parts in the case of use and production as well, in details like stamping, metal blowing, and press process. All of them bring to save the cost of tools, producing the durable products with more accurately and highly effective economy [1], [2].

Materials considered are superplastic in the detail conditions which are speed and temperature deformation that must be obtained over 200 % in elongation. Aluminum alloys have different characteristics once change alloy components. Therefore, the denaturation is dissimilar with each this type of alloy. The denaturation included two aims which are to make the fine grain in both matrix aluminum and alloy. Most aluminum alloys do not modification possess in the freezing process but instead formulation of clearly different three area microstructure in which including the outer shell of fine, and then the cylinder crystal layer and in the centre of ingots contain the rough crystals. The level of coarse grain and the length of cylinder crystals depend on pouring temperature, the gradient in mold, and the number of nucleation's formed in the crystallization process. In the view of principle, then adding the element alloy into liquid aluminum, the grain size is decreased following the rule of the solubility rate: the chemicals have a higher solubility rate, the more grain size is decreased. Alloys that contained many elements with a great solubility rate such as Cu, Mg, Zn will easily denature small grain as much as that of alloy included Si content. For instance, by using a modification for Al-Si-Mg-Cu alloy, it can obtain the grain with fine, but opposed to Al-Si with a high amount of Si content, additionally Si is hard to denature [3]–[7]. In the views of thermodynamics, the recrystallization process included two stages: nucleation and growth of nucleation. The speed of these two phases is a critical parameter in the formation of microstructure and properties of alloys. The main purpose of denaturation is to change these two factors. The modification absorption will produce a absorb layer which is cover the established crystals to protect these crystals grow up. The denaturation which produces the foreign particles: when it is doped into liquid aluminum then will react with the available elements that

are presented in the alloy to form other compounds such as oxide, sulfite, these compounds have a crystal lattice similar to the crystallization phase and consequently, it will become the crystallization nucleation for itself. B, Ti, Zr, C, Sr elements are often used to denature the solid-solution alloys. Once crystallized, these chemicals are mixed with the aluminum background to fabricate the fine phases that contained the lattice alike to the aluminum alloy's matrix phases, namely AlB_2, Al_3Ti, and Al_3Zr in the role of crystallization nuclei. This type of denaturation is often used under salt or intermetallic alloys. Phosphorus and its derivatives are the most effective denaturation in the terms of primary Si crystal grain of the hypereutectoid alloys. Phosphorus will produce the fine molecular phosphorus which possesses a lattice that is relatively similar to the silica crystal lattice; it is useful for the crystallization process of primary silica. In additional, phosphorous also forms the Cu_3P compound that benefits from the fine denaturation of the alloys. These compositions do not only increase the strength but also robust the abrasion properties under stress that relied on complicated phases in the grain boundaries. Moreover, it is also employed under intermediate alloy formation Besides the refining modification applied to make small grain size, it also has the following benefits: The additional elements for ingots sample in the ultimate stage of the crystallization process were mainly supported based on the presence of enormous nucleation centers caused by refining substances. The coarse porosities were highly increased while the micro-porosity tended to more constant distribution. Additionally, in the crystallization process, there are any gas porosities are formed will be more evenly spread in ingot samples with the fine refining denaturation. The trends of heat cracks in the crystallization process were declined to the lowest peak with the separate areas that growth inability. Consequently, the separation and cracks of grain boundaries will rarely happen. The fine structure will produce more value for the surface layer is anodized. The rough grain of ingot samples is very hard to engineer mechanical and generally, the surfaces are much less worth compared to the fine ingot sample as doing so.

The generation of super-conductivities with the fine microstructure was improved in both the strength and ductility and especially, strongly increasing the toughness thus it is hard to cause damaging brittle. Once the size is smaller causing the deformation rate is higher, and the temperature of the super-plastic effect is smaller [8], [9], [18]–[21], [10]–[17]

Due to the small grain size causing the total area of grain boundaries is larger than hindered the strong slip (increasing the strength). When the deformation at high temperature, the bond between grains becomes weaker along with high-density grains (the number of grains was adapted with the slip) that making the slip was caused more facile at the grain boundary. The slip-on grain boundary is defined as a deformation of the polycrystalline materials, that is recognized as a reciprocal movement between adjacent particles, they replace the nearby particles leading to increase the particle density follow to the tensile direction and decrease on the horizontal area-this alternative process is characteristic for super-conductivity deformation that seldom found in the normal conditions. The slip process of the particle can cause their movement along the boundary or in a boundary area. When the metal is normally deformed with a large particle size, the elongation of the test sample is often not uniform, resulting in the elongation of particles and as can be found on the cross-section of the sample, the number of particles has not decreased. In the view of high plastic deformation, the shape of each particle has less changed. If some particles are elongated, the elongation is much smaller compared with the relative elongation of tensile samples. Once the shape of the particles does not change or little change in the high plastic deformation rate this means that there is a substitution of nearby particles, leading to an increase in the grain density in the direction of tensile of sample and decrease on across area which leads to the large of deformation. In the mechanisms of the super-plastic effect, there are two mechanisms as a movement of deviation in grain and diffusion process. These two are interrelated and influence to super-plastic process of study alloy [2], [22].

Regarding the model of Ball-Hutchinson [23], the mechanism of grain boundary slip is the gradual creep and displacement of the deflection. At the recrystallization temperature, the deviation was stopped at the grain boundary due to the regulation of deformation rate from the lack of stress concentration. From the insufficient in both stress concentrations and sliding process of deflection are principles for control of deformation rate at high temperature. Mechanical – heat treating is a stage that combined heat treatment and plastic deformation, but impossible to automatically coordinate between deformation, annealing, and cooling processes. If the plastic deformation were conducted subsequent heat treating, this means that is not mechanical-heat treating but is a normal heat treatment plus mechanical pressure. For example, in the case of real plastic deformation that is cool rolling after aging, it probably results in stiffness feature which leads to increase the strength properties but without effect to the construction of microstructure as phase transformation. The plastic deformation that was performed before the heat-treating process does not affect the formation of the last microstructure; hence this combination is also not a type of thermochemical treatment.

Recrystallization annealing is defined as the main process of the ability to regulate the grain size in metal materials after deformation. In addition, after the recrystallization process has finished, the new microstructure formation is not caused by phase transformation, the main cause is the formation of the newly multi-edge particles in metals that cooled deformation. With the Al-Zn-Mg-Cu alloys, the re-crystallization temperature is elected in the range of 230 ÷ 380 ± 50°C, and (0,5÷2h) in time [24]–[26].

This article presents the results on the influence of La, Ce, and thermo-mechanical treatment on the microstructure and mechanical properties of Al-Zn-Mg-Cu alloy. The article also presents the analysis of structural change; phase transformation of this alloy in a different process.

MATERIAL AND METHODS

Material

The ingots were cut the aluminum billet into small pieces to fit in the cooking pot. The weight of each batch is about 1 1.2 kg of aluminum billet. After cutting, the workpiece will be cleaned of dust and dried at a temperature of 200°C to remove moisture remaining on the surface of the workpiece. The addition of pure metal is required to achieve the desired batch composition. The mixing ratio has been calculated in advance. Pure Zn and Mg are chopped, dried together with aluminum billets before being used for melting.

The modification elements used here are rare-earth with the main ingredients being La and Ce. After melting, the alloys get in the mold and analyzed chemical composition. The chemical composition was present in **Table 1**.

Table 1 Chemical composition

Sample	Zn	Mg	Cu	Si	Fe	Mn	Cr	La	Ce	Al
M1	5,4	2,2	1,33	≤0,34	≤0,19	0,165	0,018	-	-	Bal.
M2	6,1	2,2	1,74	≤0,34	≤0,19	0,165	0,018	0,155	0,21	Bal.

Methods

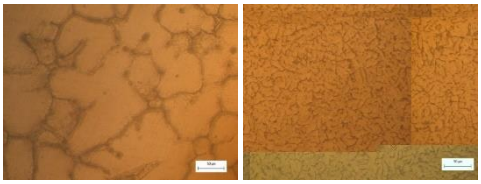
The ingots are conducted homogeneous annealing at 480°C within 16h to ensure removal of casting microstructure. After the homogeneous process, the patterns are rolled from the thickness of 4 mm to 2 mm. Next, all the samples were recrystallized annealing at 400°C in 1h15.

The microstructure of these patterns is investigated by using optical microscopic, SEM, EDS, and X-ray diffraction and mechanical test (mainly determined the deformation level).

The tensile test is applied on the patterns that determined deformation degree at 400°C, the results were extracted in elongation value

RESULTS AND DISCUSSION

From the micro-optical analysis results (**Fig. 1**): the samples with La, Ce show a smaller grain size compared to the samples without one as race earth.

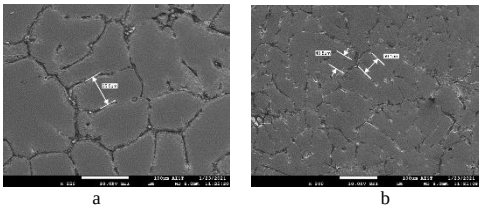


a b

Fig. 1 Microstructure of sample after casting: *a* – M1 sample; *b* – M2 sample

The finding on the grain size of the race earth samples has eight levels, while the samples without race earth composition possess at six-level (As the ASM standard). Thus, once the alloys are added to the race substance, the grain size witnessed a considerable decrease compared to that of without race earth compound. This is can be explained by the role of race earth in the fine grain of Al-Zn-Mg-Cu alloy. Making the grain allocation more uniform than without La, Ce and no longer existing the high gap of grain size. By using the optical-microscope analysis cannot find the structure and intermetallic phases of RE with elements in alloy's studying.

In **Fig. 2**, while the distance between tree branches of samples without LA, Ce was approximately 65 µm, the sample with La, Ce decreased to (10-50)µm. it is worth noting that due to the role of the RE phase that is decreased the size of tree branches.



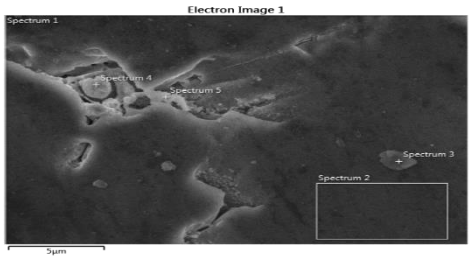
a b

Fig. 2 Microstructure of sample after casting by SEM: *a* – M1 sample; *b* – M2 sample

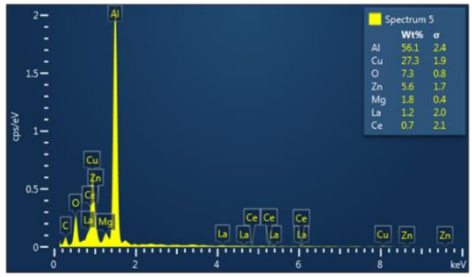
Therefore, the alloys with RE compound exhibited the Al intermetallic phase combined with the existed elements, as shown in the SEM and XRD figures. Additionally, to further improve that can be used TEM method to determine those intermetallic phases and explain in detail the roles.

From the optical-microscope result of alloys, patterns exhibited the brown color of matrix phase as Al phases, and the black color parts predicted as the intermetallics of Zn, Mg, Cu metals and possible to presence of race earth compounds mixed into the black color phases.

That improvement above was confirmed by SEM and EDS analysis at the grain boundary of post-casting as follows below.



a



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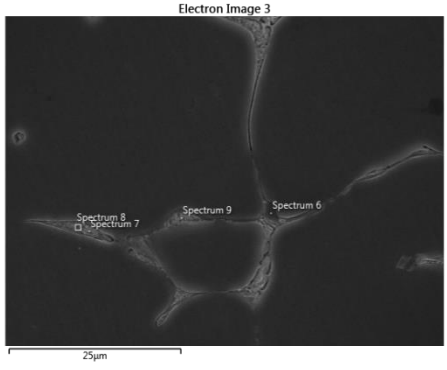
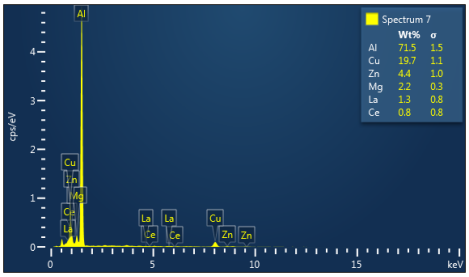


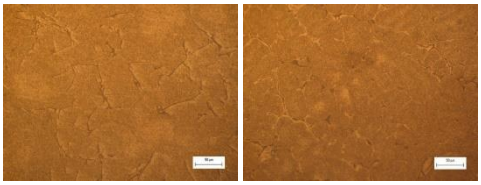
c



d

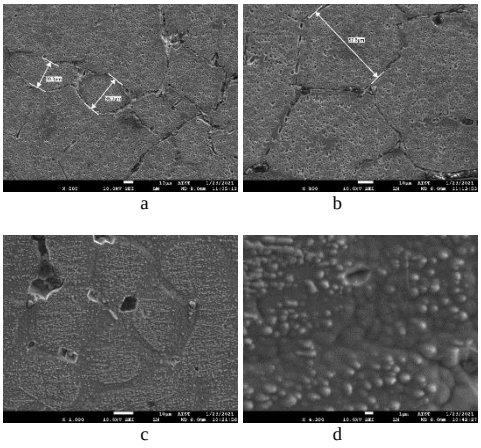
Fig. 3 EDS of M2 sample: *a* – SEM of spectrum 5; *b* – EDS of spectrum 5; *c* – SEM of spectrum 7; *d* – EDS of spectrum 7

Fig. 3 shows the typical peaks of consisting La and Ce in alloys at the grain boundary, manifesting characteristics peaks at 5 and 7. The alloys gathered at the grain boundary that is linked to other elements to form intermetallic phases which is stop the growth of grains after adding the rare earth compound.
After homogeneous annealing at 480°C within 16h shows the grain size has higher than the grain size of casting samples, as shown in **Fig. 4**. In the terms of alloys consisting of La and Ce, the grain level is 7 after homogeneous, while without La, Ce is just over 5. It is worth noting that the microstructure at the casting state was eliminated after homogeneous annealing.



a b

Fig. 4 Microstructure of samples after homogeneous annealing (OM): *a* – M1 sample; *b* – M2 sample



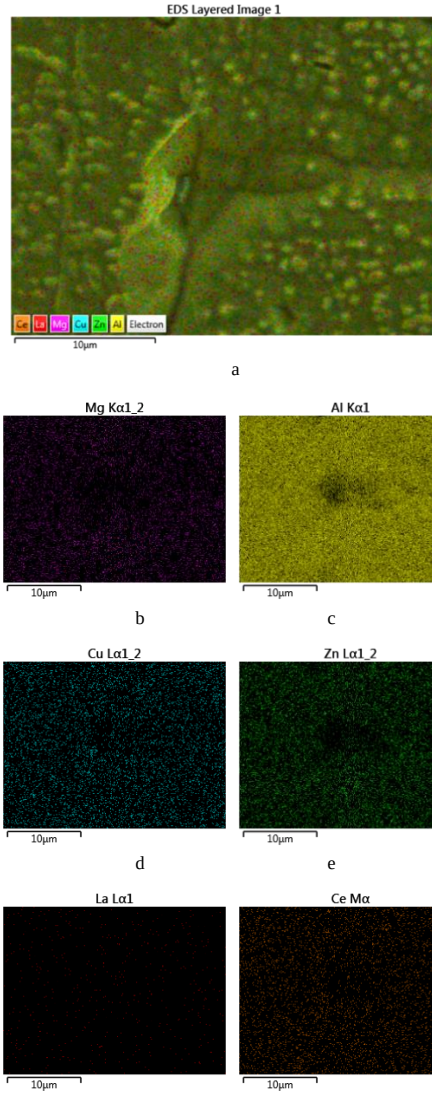
a b

c d

Fig. 5 Microstructure of samples after homogeneous (SEM): *a* – M1 sample (x500); *b* – M1 sample (x800); *c* – M2 sample (x1000); *d* – M2 sample (x4300)

According to the alloys without La, Ce, the grains are not uniform and relatively large at above 57 µm in **Fig. 5*b***, and some grains at 20-30µm range in **Fig. 5 *a***. By contrast, with the alloys, consisted La, Ce, there is a medium grain size of 30µm at the lowest resolution and relatively uniform grain sizes in **Fig. 5*c, d***. Moreover, the higher resolution shows the uniformation and relatively small grains

Fig. 6 shows the very uniform of elements on the whole area after homogeneous annealing, which is compared with the casting sample. There is La, Ce concentration at the grain boundary. Thereby, the elements were uniformly solid solutions on the surface of homogeneous samples.



a

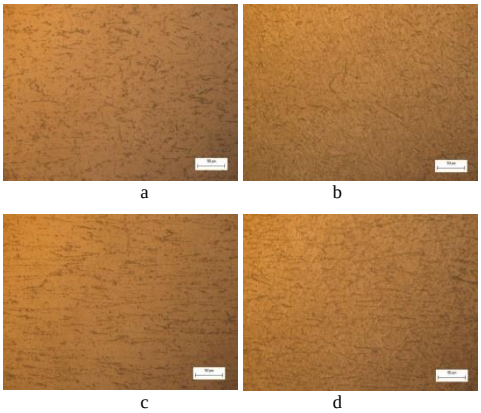
b c

d e

f g

Fig. 6 Element spectrum of M2 sample after homogeneous annealing by Mapping: *a* – mapping all elements; *b* – mapping of Mg; *c* – mapping of Al; *d* – mapping of Cu; *e* – mapping of Zn; *f* – mapping of La; *g* – mapping of Ce

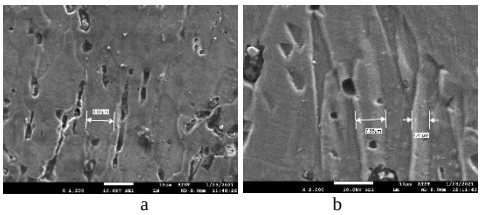
In **Fig. 7**, the grain size is smaller after homogeneous annealing; with the samples with La, Ce that is evenly distributed of microstructure and without black color phases compared with samples which not included la, Ce. These characteristics are consistent with the analytical results which will be discussed later.



a b c d

Fig. 7 Microstructure of samples after deformation: *a* – M1 sample (Perpendicular to rolling direction); *b* – M2 sample (Perpendicular to rolling direction); *c* – M1 sample (rolling direction); *d* – M2 sample (rolling direction)

After deformation shows the sample with La, Ce is the smaller distance of vestigial deformation than the sample without La, Ce, as shown in **Fig. 8**.

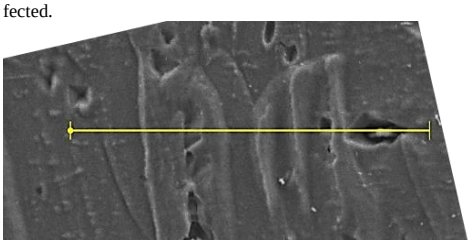


a b

Fig. 8 Microstructure of samples after deformation (SEM): *a* – M1 sample; *b* – M2 sample

The measurement of EDS lines was indicated the main elements in an alloy that are still existed the uniformity of these elements (**Fig. 9**).

From **Fig. 10**, samples M1 and M2, after deformation and annealing recrystallize, the grain size becomes more uniform. However, with sample M2 the average grain size of the sample was 9.4 µm while with sample M1 the average particle size was above 15 µm. Thus, it can be observed that the influence of La and Ce will greatly affect the grain size. With such a change in particle size, the mechanical properties of the sample will be affected.



a

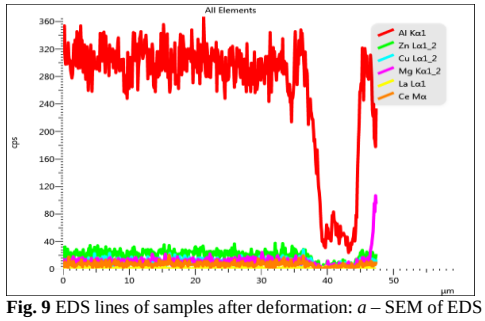
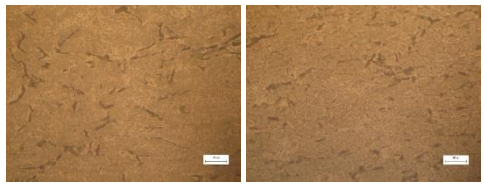


Fig. 9 EDS lines of samples after deformation: *a* – SEM of EDS line; *b* – EDS line



a b

Fig. 10 Microstructure of sample after deformation and recrystallization annealing: *a* – M1 sample; *b* – M2 sample

Table 2 shows the different degrees at the highest deformation level of the samples with and without La, Ce. While the elongation value of M1 is 11.58 %, the samples with 4 % modification (M2) are 31.92 % consistent with an increase of 63 %. This may be attributed that although the grain size is not largely decreased the deformation level is highly effective. The tensile test result indicates that the effective denaturation process contributed to a significant increase in the deformation degree of alloys.

Table 2: The result about ductility and strength of samples

Samples	Ductility ε (%)	Limit strength σ_b (MPa)
M1	11.58	39.5
M2	31.92	58.3
M1-deformation (M1-1)	74.9	48.1
M1-deformation- recrystallizion annealing at 400°C in 1h15(M1-2)	82.4	78.1
M2-deformation (M2-1)	121	47.8
M1-deformation- recrystallizion annealing at 400°C in 1h15(M2-2)	140	104

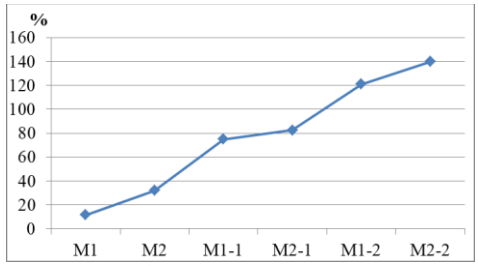


Fig. 11 Diagram of ductility (%)

Fig. 11 based on the results of samples which are deformation combined with recrystallization annealing alloys are higher in elongation than the samples with La, Ce, in detail at 140 %. These results are consistent with the research presented above.

In addition, the grain size of deformation combined with recrystallization heating alloys becomes smaller once compared with the samples that no both denaturation and deformation.

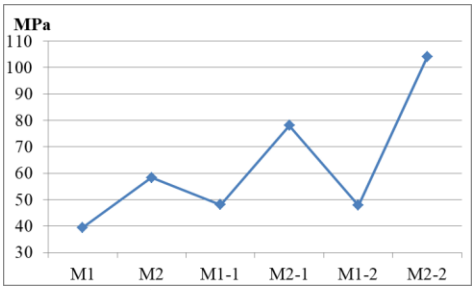


Fig. 12. Diagram of limit strength (MPa)

Fig. 12 analysis of strength limit results shows that: The strength limit has the highest value when the M2 is deformed and recrystallization annealing. This value is consistent with the results about microstructure.

CONCLUSIONS

In this study, the article is determined the transformation of the morphology and structure of Al-Zn once with and without La, Ce elements. Moreover, the change of microstructure of alloys is also presented in this work. These results in mechanical properties after homogeneous heating, deformation, and recrystallization annealing indicate the increased inductivity of the study's alloy. In addition, the alloys based on Le, Ce also attributed to an increasing deformation.

Acknowledgements: Thanks to the support from the School of Mechanical Engineering - VMU, School of Materials Science And Engineering – HUST, and experiment facilities from Prof. Tran Duc Huy's lab. This is an appreciated acknowledgement for us to complete this study.

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