ICMIEE22-009

Analysis of Microstructure and Mechanical Behavior of Zamak 2 Alloy on Precipitation Hardening

Umma Habiba Afia^{l, *}, Fahim Khan^l, Jahirul Islam^l

¹Department of Materials Science and Engineering, Khulna University of Engineering & Technology, Khulna-9203, BANGLADESH

ABSTRACT:

Zinc alloys contain significant qualities that make them appropriate for industrial usage, such as a low melting point, nontoxicity, and high corrosion resistance. Zamak 2 alloy has 3.9 percent aluminum, 2.9 percent copper, and the rest zinc. The presence of copper in this alloy causes the precipitate (CuZn4) phase to form, which improves mechanical qualities. However, more than 1.25 wt.% copper causes dimensional instability, which can be reduced by heat treatment. The main focus of this experimental report was on precipitation hardening and comparison of characteristics between casted Zamak 2 sample and heat treated Zamak 2 samples. This technique achieves strengthening by generating particle dispersion, which acts as a barrier to dislocation movement. Here the magnitude of strengthening is determined by the interaction between dispersion particles and dislocations. Simultaneously, the micro-structural changes and mechanical characteristics augmentation of Cu doped alloys were also compared to the non-heat-treated alloy. Micro-structural variations and mechanical characteristics were considerably altered after and before precipitation hardening.

Keywords: Precipitation hardening, Aging, Heat Treatment, Dislocation, Dendrite.

1. INTRODUCTION:

Alloys are composed of two or more chemical elements, at least one of which is a metal, that are combined to form a mixture.[1] There are two possible outcomes for the alloying element: it may either be dispersed throughout the crystal lattice sites of the host element and result in a solid solution, or it can produce various phases that manifest themselves as particles in a "matrix."[2] Zinc alloys have some significant properties like low melting point, environment friendliness as well as good corrosion resistance for which it is supposed to have great application in industrial work. Variation of zing casting alloy depends upon composition as hypereutectic alloys containing 5.1 wt. % aluminum, likewise eutectic with alloys 5.1 wt. % aluminum and last of all and hypoeutectic alloys less than 5.1 wt. % aluminum. Zinc alloys containing less than 5.1 aluminum that means hypoeutectic alloy is known as ZAMAK alloy.[3] Zamak alloys exhibit good fluidity and formability during casting process. For which casting process is chosen to make specimen of Zamak 2 alloy (ZnAl₄Cu₃). Here composition of Zamak 2 alloy is 3.9 % Al, 2.9% Cu and rest of Zinc so it is highly preferable for heat treatment. For this study gravity casting is approached to make specimen.[4] Zamak 2 alloy exhibit best mechanical properties like good tensile strength and creep resistance and hardness. In this alloy presence of copper leads to precipitate ε (CuZn₄) phase which increases mechanical properties but more than 1.25 wt. % of copper becomes reason of dimensional instability which could be minimize via heat treatment.[5]. In this experimental report precipitation hardening is main concern. At this process strengthening is obtained by producing particle dispersion which works as an obstacle to dislocation movement [Precipitation-hardening of metals].[6]

Interaction between dispersion particles and dislocation determine the magnitude of strengthening. Precipitation hardening is carried out by two steps like basically at first stage alloy reach high temperature and becomes liquid then it is quenched again rises to little bit high temperature to get homogeneous send phase dispersion.[7] So at this paper microstructure and mechanical properties of casted Zamak 2 alloy have been investigated as well as effect of large amount of copper in Zamak 2 alloy through precipitation hardening has been observed. At last the effect of precipitation on microstructure and mechanical properties upon Zamak 2 alloy also has checked out.

2. METHOD:

This experimental procedure was focused on zinc alloys. Among zinc alloys Zamak are best for their castablity and precipitation hardening. For making Zamak alloy at first raw materials were collected. Here the percentages of elements in Zamak 2 are given below:[8]

Table 1: Zamak 2 composition with compounds.

Zn	Al	Mg	Cu
Balance	4.197	.04	2.79

2.1 Working procedure:

This experiment procedure is classified into some particular sections like melting, casting, precipitation hardening followed by testing.

2.1.1 Casting

At first a sand mould was prepared. Due to unavailability of die casting a sand mould casting was chosen for doing the casting operation. For this at first a sample with

E-mail addresses: afia27037@gmail.com¹, fahimkhanprionto@mse.kuet.ac.bd², jahirul@mse.kuet.ac.bd³.

^{*} Corresponding author. Tel.: +88-0179995650

desired shape and dimensions was selected first. Moulding typically was prepared by following steps:

- Preparing the consolidated sand mould around a pattern held within a supporting metal frame, and
- Removing the pattern to leave the mould cavity with cores

Then sample was placed in the drag and filled up with sand. After that this specimen was pulled out and the mould was ready. The mould was preheated at 250° C for 10 minutes that sand's particle can cluster together proper way which increases the strength. Melting was accomplished using electricity in the foundry shop. As Zamak 2 alloy is a hypoeutectic alloy and its melting point is around 450°C.[9] So raw materials were melted around this temperature. At last molten metals were solidified. And during solidified, there occurs shrinkage at above that's why extra materials are provided. Then from the mould the casted part was collected.

2.1.2 Precipitation hardening:

The samples were placed in a heating chamber to be heated even more thoroughly. For this experiment, a total of four specimens were created. One casting sample is retained for the purpose of measuring raw data. The remaining three specimens will be subjected to additional heat treatment. Because of the segregation of the alloying components in as-cast alloys, there is a non-uniform distribution of solute in the microstructure.[10] This phenomenon, also known as coring, has important implications in precipitation hardening alloys, where final solidification of a single phase is expected according to the phase diagram, but in practice a huge number of solute atoms segregate to grain boundaries during solidification, resulting in a hardening effect.[11] This precipitation prevents austenite from recrystallizing even more. [12] Alloys were homogenized at 250 C to achieve a homogeneous microstructure since there would be diffusion of alloying elements from the grain boundaries. In order to produce homogeneity, solute atoms must be uniformly distributed throughout the matrix, which is accomplished by the use of a homogenization heat treatment. Homogenization was carried out at 250C for a total of 30 minutes. The alloy piece was removed from the furnace after it had been homogenized and quenched in water for several minutes. Those have been placed in the oven at a temperature of 250 ° C. For heating, an electric oven was employed, which was completely isolated from the surrounding environment. For this there is no chance for oxidizing. Sample was kept in air. As three sample was for further experimental work. So basically, the variations occur at natural aging time. Like for first sample it was kept in air environment for one week. And then second one was kept in nature for two weeks. Next two piece were kept at nature for six and eight weeks.

Then sample was cut into pieces and made prepare for further experiment. At first sample was prepared for microstructure observation. And for this mounting of small pieces, course grinding, medium and fine grinding, mechanical polishing followed by etching process were done step by steps. At last prepared sample was kept under electron microscope. A metallurgical microscope is equipped with a lens system (objectives and eyepiece) that enables it to reach magnifications ranging from 25X to 1000X.

2.2 Testing

Then further mechanical tests were proceeded. Those are tensile strength test, impact test, hardness test. Tensile test was carried out by manual tensile testing machine. From this machine out of it got as load to break the sample. After that area is calculated. From area and load tensile strength is calculated. Then Brinell hardness test is used for larger samples in materials having a coarse or inhomogeneous grain structure.[13] Brinell hardness is determined using the Brinell hardness test method. For measuring hardness in this experiment Brinell harness without doubt best option. Brinell testing commonly employs a very high-test load (3000 kgf) and a 10mm diameter indenter to average out most surface and subsurface irregularities. For impact test sample specification according to ASTM A370 was length was 55mm ang height and width was 10 mm and 10mm.[14]

3. RESULT AND DISCUSSION

3.1 Microstructure analysis

Here the microstructure of the sample was taken after casting and after precipitation hardening.

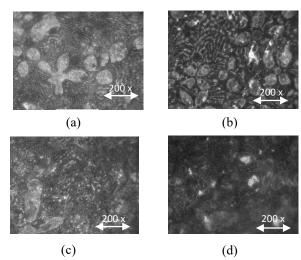


Figure 1: Microstructure of (a) casted sample (b) after precipitation hardening and 1 week of aging (c) 6 weeks of aging (d) 8 weeks of aging.

In figure one four microstructure is given which were taken by electron optical microscope at 200x, 400x and 1000x. All the microstructures are given in figure 1 and from those images all the grain boundaries can be observed well. From the microstructures it can be seen

that casted alloys have a dendrite matrix corresponding to a primary zinc-rich phase (η -phase), this is known as η primary dendrites. Also, there is one eutectic interdendritic microconstituent, and one eutectoid isolated region which is known as (η + eutectoid) region.

In second microstructure is of sample which is precipitation hardened and air aged for one week. It is clearly seen that microstructure is more homogenized than first one. And phases are evenly distributed. And the structure is formed by η phase and Aluminum rich α phase. Black points occurring in the dendrites of η phase and ϵ phase. Here η phase are constitutes of precipitations of ϵ phase also called CuZn₄.

And (c) and (d) microstructures are respectively specimen of air aged for 6 weeks and 8 weeks. They also possess the homogeneous microstructure.

The reason behind homogeneous precipitate structure is sample was heated at high temperature that is 250°C and cooled down by normal water. For this when the sample is cooled down ϵ phase that means $CuZn_4$ can't be solute evenly with sudden decreases of temperature. So, it becomes over saturated. And for this reason, precipitation of ϵ phase occurs in η phases.

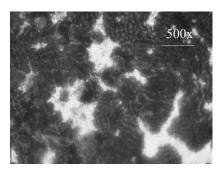


Figure 2: Microstructure of precipitation hardened sample at magnification of 500x.

From above figure dendritic structure can be understood clearly. Black portion is Al- riche phase whereas white portion is Zn- rich phase with CuZn₄ precipitates.

3.2 Tensile test

Tensile properties can explain the effect of precipitation hardening on the mechanical characteristics of Zamak 2 alloy. Here the applied force divided by the initial cross-sectional area results in engineering stress, also known as nominal stress. [15] Due to length constraints, the tensile test was performed manually in this case. Total 4 specimens were tested. Among them one was tested after casting. And rest of them after precipitation hardening which differ strength data. Among three specimens two were over aged and the tensile test data table shows respective changes.

Clearly, the tensile strength of the material increased gradually after precipitation hardening, as can be seen in the following table:

Table 1: Elongation and tensile properties of one casted Zamak 2 and three precipitation hardened samples.

Alloy	Tensile	Elongation (%)
	strength	
	(MPa)	
Zamak-2	210	6
(casting sample)		
After 1 week of	225	7
aging		
6 weeks of	217	6.9
aging		
8 weeks of	216	6.9
aging		

This occurs for the precipitation of the $(CuZn_4)$ phase in the phase during the phase. As a result, dislocation movement is hindered, resulting in an increase in ultimate tensile strength. After precipitation hardening, elongation increases progressively in proportion to the variance in tensile strength and elongation. However, the rate of increase in elongation is somewhat slow for the remaining two samples. Because they have been aged for a long time.

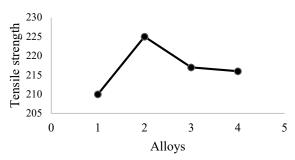


Figure 3: Comparison of tensile strength between casted Zamak 2 alloy and precipitation hardened alloys.

Percent elongation is a measure of a material's ability to deform plastically and elastically up to fracture. Percent elongation is one approach to quantify and measure a material's ductility. So it can be said that materials Zamak 2 alloy's ductility is increased by precipitation hardening. And with aging time ductility is also increased.

3.3 Hardness

From the graph (figure 4) it is clearly showed that hardness is increased after precipitation hardening of one-week aging. The reason behind is that when sample was quenched at water then phase CuZn₄ was precipitated at Zinc rich phase and after one week of aging the phase was unsaturated and which was reason for increases of hardness. So, the initial hardening arises due to an increase in dislocation density and due to precipitate-dislocation interactions

With the time the hardness of the precipitated sample of 6 weeks of age hardened and 8 weeks of age hardened have to increase from one week of aging. But instead it decreased. This may occur due to precipitate phase which

forms as finely dispersed particles of composition. With increasing aging time precipitated phase $CuZn_4$. ϵ may become equilibrium. And this may occur in zinc alloy exhibit dimensional changes and natural aging by which it's hardness and tensile strength reduces.[15]. Table 2 shows variations of hardness among casted alloy and three casted and precipitated Zamak 2 alloys.

Table 2: Hardness variation with casted Zamak 2 alloys and precipitation hardened alloys.

Alloy	Indentation depth	Hardness
	(mm)	(BHN)
Casted Zamak	3.1	77.536
alloy		
After 1 week	3.04	80.70
of aging		
6 weeks of	3.07	79.09
aging		
8 weeks of	3.08	78.57
aging		

Also, continuation of the aging process ends up in a discount in hardness and strength properties, called over aging. For better understanding of hardness variations bar chat has been included.

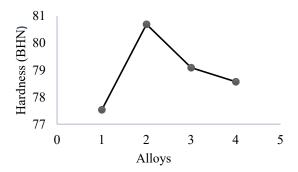


Figure 4: Hardness comparison among casted Zamak 2 alloy without heat treatment and Zamak 2 alloys with heat treatment

It has been said that the combined action of fatigueinduced dislocations and vacancies may induce over aging, i.e., the replacement of the metastable precipitates by more stable and more widely spaced precipitates.

Yet the hardness of precipitation hardening treated alloy are greater than casted alloy.

3.4 Impact test

Likewise, to other test this test also done for four samples. One sample is tested aster casting. And the rest three samples are tested after precipitation hardening. The results are given below;

The ability of a material to withstand impact and fracture shows its ability to inhibit crack growth. Toughness is typically associated with plasticity: greater plasticity is associated with greater toughness. Even though there is normally a trade-off between strength and ductility, the relationship between strength and toughness is not a straight forward reciprocal one.

When it comes to structural materials, it is essential to have both high strength and high toughness at the same time in order to handle more weight while preventing catastrophic failure.

Table 3: Comparison of impact strength and absorbed energy of experimented samples.

Alloy	Impact strength (Jcm ⁻²⁾	
Casted Zamak alloy	65.625	
After 1 week of aging	64.042	
6 weeks of aging	64.843	
8 weeks of aging	64.843	

Figure 5 : Comparison chart of impact strength among experimented alloys. (Casted alloy and heat-treated alloys)

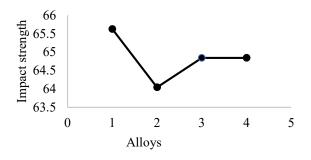


Figure 4.6: Comparison chart of impact strength among experimented alloys. (Casted alloy and heat-treated alloys)

Among the techniques used to determine impact toughness of materials under high strain rates, the Charpy impact test is the most widely used. It has been found that the specimen size, notch size, internal flaws (inclusions, porosity), microstructure, temperature, and other factors influence the impact toughness.

Here after precipitation the grain and dendrites become small then before which is reason behind of decreasing toughness. Also, there a precipitated particles of Cu phase in Zn rich matrix. Which also reduces toughness for one week of aged sample.

Due to over aging and precipitated particles becomes stable and wide spaced that's why toughness increased and last two sample are almost same.

4.CONCLUSION.

The purpose of this experimental study was to investigate the effect of precipitation hardening on one of the most widely used zinc alloys. The hardening treatment in this case was carried out using Zamak 2 alloy. The experimental work was expected to be carried out in a structured manner. When casting four samples,

one sample was maintained for comparison, and the other three were heated and kept for air aging after they were finished. The following are the conclusions obtained as a result of these experimental studies:

- In the casted Zamak 2 alloy, the microstructure is dendritic, with dendrite phases formed by the presence of a zinc-rich phase in an Al-rich phase. Primary dendrites are found in zinc-rich phases in this region.
- After precipitation hardening dendritic phase or laminar phase becomes more conjugated. And CuZn₄ phase precipitated as ε phase. This can be understood from microstructure of one-week aging. But due to unexpected reason sample couldn't examine after three and four weeks of aging. That's why there occur over aging, due to this reason microstructure of those sample is not cleared.
- Variation of tensile test assured that after precipitation alloys strength rises after hardening process. But as said before due to over aging tensile strength drops. As well as zinc alloys exhibit unstablity for this its strength drops gradually. So, this can be overcome by increasing heating duration. So, when zinc alloys like Zamak 2 will be used as a casted part of any application where higher tensile strength is needed there before application Zamak part can be precipitation hardened. So, it will increase its strength.
- Likewise, tensile strength alloy's hardness also risen after precipitation hardening. But drops for 6 and 8 weeks of aged sample.
- Due to precipitation hardening stress concentrated at those particles. Due to this after precipitation hardening it can't absorb more energy and becomes brittle. Due to stabilization of metastable phase or precipitated phase its impact strength rises.

References

- [1] A. P. Eccles, "THE EVOLUTION OF AN ALLOY MELBOURNE AUSTRALIA."
- [2] W. Pfeiler, Alloy physics: a comprehensive reference. Wiley-VCH, 2007.
- [3] F. Costa Da Silva, K. Kazmierczak, C. Edil Da Costa, J. C. G. Milan, and J. M. Torralba, "Zamak 2 Alloy Produced by Mechanical Alloying and Consolidated by Sintering and Hot Pressing," Journal of Manufacturing Science and Engineering, Transactions of the ASME, vol. 139, no. 9, 2017, doi: 10.1115/1.4037181.
- [4] S. Krýslová and I. Nová, "the Wall Thickness Influence of Casting and Mould on," Metal, pp. 1–5, 2008.
- [5] B. J. Li and C. G. Chao, "Aging kinetics of heattreated Zn-4Al-3Cu alloy," Scripta Materialia,

- vol. 41, no. 2, pp. 143–147, 1999, doi: 10.1016/S1359-6462(99)00130-X.
- [6] W. L. Fink, "Precipitation hardening," Journal of Applied Physics, vol. 13, no. 2, pp. 75–83, 1942, doi: 10.1063/1.1714842.
- [7] I. Nová, I. Nováková, and J. Machuta, "Precipitation hardening of zinc alloys castings Available: https://www.researchgate.net/publication/28855 4566
- [8] M. Gelfi, E. Bontempi, A. Pola, R. Roberti, D. Rollez, and L. E. Depero, "Microstructural and mechanical properties of zinc die casting alloys," Advanced Engineering Materials, vol. 6, no. 10, pp. 818–822, Oct. 2004, doi: 10.1002/adem.200400087.
- [9] A. Azizi and G. G. Haghighi, "Fabrication of ZAMAK 2 alloys by powder metallurgy process," International Journal of Advanced Manufacturing Technology, vol. 77, no. 9–12, pp. 2059–2065, Apr. 2015, doi: 10.1007/s00170-014-6611-z.
- [10] Z. Guo and W. Sha, "Quantification of Precipitation Hardening and Evolution of Precipitates," 2002.
- [11] A. J. Ardell, "Precipitation Hardening."
- [12] Khan F. Book Chapter "Phase Transformation in Micro alloyed Steel" of "Engineering Steels and High Entropy-Alloys", ISBN 978-1-78985-948-
- [13] "A theoretical study of the Brinell hardness test," Proceedings of the Royal Society of London. A. Mathematical and Physical Sciences, vol. 423, no. 1865, pp. 301–330, Jun. 1989, doi: 10.1098/rspa.1989.0056.
- [14] "Standard Test Methods and Definitions for Mechanical Testing of Steel Products 1", doi: 10.1520/A0370-16.
- [15] Khan F., Rashed H. M. M. AL., High Temperature Properties of Common Structural Steels, ICMIME, RUET: 374-379, 2019
- [16] E Gervais, R. J. Barnhurst, and C. A. Loong, "An Analysis of Selected Properties of ZA Alloys."