

Structure and properties of alloys of the Mg-Al-Zn system

L. Čížek ^{a, *}, M. Greger ^a, L.A. Dobrzański ^b, I. Juřička ^c, R. Kocich ^a, L. Pawlica ^a, T. Tański ^b

VŠB-Technical University of Ostrava, 17. listopadu 15, 708 33 Ostrava, Czech Republic
Division of, of Materials Processing Technologies, Management and Computer
Techniques in Materials Science, Institute of Engineering Materials and Biomaterials,
Silesian University of Technology, ul. Konarskiego 18a, 44-100 Gliwice, Poland
Ferona- Steel Servis Centrum, Hradec Králové, Czech Republic

* Corresponding author: E-mail address: lubomincizek@vsb.cz

Received 01.12.2008; published in revised form 01.02.2009

Properties

ABSTRACT

Purpose: In the following paper there have been the structure and complex of mechanical properties of magnesium alloys presented which requires very often knowledge of elastic-plastic properties at elevated temperatures. These properties are connected with microstructure that is influenced by metallurgical and technological factors and conditions of exploitation. The influence of used method ECAP for superplasticity will be investigated.

Design/methodology/approach: The tensile test in dependence on temperature of magnesium alloys was based on investigation of mechanical properties. The following results concern light and scanning microscopy for metallographic and fracture analyses of alloys after testing were used.

Findings: Objective of this work consisted in determination of changes of elastic-plastic properties of magnesium alloy AZ91 as cast state and after heat treatment in dependence on temperature, including investigation of fracture characteristics. It was confirmed that during heating used alloy as cast state at chosen temperatures there occurs partial dissolution of minority phases. Homogenisation of microstructure is, however, accompanied by simultaneous forming of inter-granular non-integrities, which is unfavourable from the viewpoint of strength and plastic properties, especially at higher temperatures. Failure occurs practically at all temperatures basically by inter-crystalline splitting along the boundaries of original dendrites. At temperature testing near melting point of alloy the interdendrite areas melting were observed. After application ECAP the effect of superplasticity (200-400%) was occurred.

Practical implications: The results may be utilized for a relation between plastic and strength properties of the investigated material in process of manufacturing and design of these materials.

Originality/value: Complex evaluation of properties magnesium alloys at higher temperatures namely for explanation of fracture mechanism near the melting point. The possibility of supperplasticity effect of Mg-Al alloys was not presented yet.

Keywords: Mechanical properties; Magnesium alloys; Tensile test at elevated temperatures; Fracture characteristics

Reference to this paper should be given in the following way:

L. Čížek, M. Greger, L.A. Dobrzański, I. Juřička, R. Kocich, L. Pawlica, T. Tański, Structure and properties of alloys of the Mg-Al-Zn system, Journal of Achievements in Materials and Manufacturing Engineering 32/2 (2009) 179-187.

<u>1.Introduction</u>

Magnesium alloys represent wide group of structural materials with specific properties, which are extensively used particularly in aerospace, but also at manufacturing of optical devices and instrumentation, in automotive and textile industries [1, 3, 6, 9, 10, 15, 16, 18, 19]. The alloy AZ91 represents the most frequently used type of magnesium alloys with good castability, with advantageous combination of strength, plastic and corrosion properties.

The material selection is preceded by the analysis of many factors like: mechanical, design, environmental, urbanization, recycling, cost, availability, and weight related issues, which may change the existing conditions and emerge the needs resulting from the supplier-customer relation [10,16]. The effort to decrease the weight of products becomes an important challenge for designers and process engineers. The excessive weight verifies a significant extent the possibilities of employing particular material groups.

Contemporary materials should possess high mechanical properties, physical and chemical, as well as technological ones, to ensure long and reliable use. The above mentioned requirements and expectations regarding the contemporary materials are met by the non-ferrous metals alloys used nowadays, including the magnesium alloys.

The designers closer and closer cooperate with magnesium alloy manufacturers, which is a good example of the fact that currently about 70% of the magnesium alloy castings are made for the automotive industry. Lowering car weight by 100 kg makes it possible to save 0.5 l of petrol per 100 km. It is anticipated that in the following years the mass of castings from magnesium alloys in an average car will rise to 40 kg, internal combustion engines will be made mostly from the magnesium alloys and car weight will decrease from 1200 kg to 900 kg [8].

Magnesium alloys has been used for a wide variety of applications, namely from the reason of their low density and high strength-to-weight ratio. Low inertia, which results from its low density, is advantageous in rapidly moving parts, for example automobile wheels and other automobile parts [6, 7, 13, 14, 17].

The basic magnesium alloys include ones which contain manganese, aluminium, zinc, zirconium and rare-earth elements which allow obtaining suitable properties. Manganese does not cause any increase of tensile strength, however, it does slightly increase the yield point. It also brings about an increase of resistance to the action of sea water. The quantity of manganese in magnesium alloys is limited by its relatively low solubility in magnesium.

Zirconium is added to alloys which contain zinc, rare-earth elements, thorium and their combinations, for the purpose of structure refinement. Scope of utilisation of foundry magnesium alloys is continuously being extended, so if we want to operate as competitive producers, it is necessary to investigate very actively properties of individual alloys, optimise their chemical composition, study issues of their metallurgical preparation, verify experimentally their casting properties and conditions of successful casting of castings by individual methods, including heat treatment, forming and others specials methods of processing. The type of heat treatment depends namely on the chemical composition of the alloys [7].

Apart from the commonly used Mg binary alloys, ternary alloys (eg. Mg-Al-Zn, Mg-Al-Si) are very widely used, as well as their more complex ones (Fig. 1).

Magnesium alloys are subjected to heat treatment mostly for the purpose of improvement of their mechanical properties or as an intermediary operation, to prepare the alloy to other specific treatment processes (for example the ECAP method) [11, 12]. A change of the heat treatment basic parameters has an influence on a change of the properties. Annealing significantly decreases the mechanical properties and causes improvement of plastic properties, thus facilitating further treatment.

Complex evaluation of magnesium alloys requires very often knowledge of elastic-plastic properties at increased temperatures.

Goals of the work are determination of changes of elastic-plastic properties of magnesium alloy AZ91 in dependence on temperature, including investigation of structure changing and fracture characteristics.

Next objective of this work is investigation of possibilities the ECAP methods applications on magnesium alloys and determine conditions for observation.

2.Used material and _____experimental technique

Investigation was made with use of cast plates (size 10x20x150 mm) of model alloy of the "Electron" type with the following chemical composition (weight %): Mg, Al-8.25, Zn-0.63, Mn-0.22, Si-0.035, Cu-0.003, Fe-0.014, Be-0.002, Zr-0.002, rest Mg.

Samples for tensile test had a form of bar with length 115 mm, diameter 6 mm, in central part the diameter was reduced to 4 mm in the length of 30 mm.

Samples as cast state - signed A and after heat treatment T4 (after ASTM) - signed A1 were used. Conditions at heat treatment: pre-heating $375^{\circ}C/3h \rightarrow$ heating $415^{\circ}C/18h$, cooling on air.

Samples for ECAP method after application of heat treatment T4 and rolling were used for next investigation. Size of sample for ECAP method was 8 x 8 x 50 mm.

Testing of mechanical properties was made on testing machine INOVA TSM 20. Temperature range of the equipment is up to 800°C. Heating to the required temperature is realised in 3 stages in argon atmosphere.

Testing of mechanical properties of samples after ECAP method on INOVA TSM 50 was realized.

Samples for tensile test after ECAP had a form of plate and measured size of sample was $3 \times 12 \times 1.5$ mm. Total length of sample was 40 mm.

3. Results of tests and discussion

Results of measurement of elastic-plastic properties of cast alloy by tensile test in dependence on temperature are summarised in the Fig. 1. and alloy after T4 in Fig. 2.

Properties

This Figure shows that values of R_m swiftly decrease with increasing temperature of the test. In other measured values there was registered initial growth with indistinctive maximum in temperature zone of approx. 250°C for work to rupture, and approx. 300°C for elongation and reduction. After achieving of the maximum there follows sharp fall, at the highest temperatures the achieved values are mostly lower than the values at the temperature of 20°C.

In Fig. 2. values of R_m at ambient temperature is significant higher than in the case of initial alloy. The temperature dependence is similar, but decreasing of R_m is started at lower temperature than in the case of initial alloy. This phenomenon is probably connected with dissolvability of intermetalic phase $Mg_{17}Al_{12}$ in the case of initial alloy during of testing.

Tensile test of samples after ECAP method was made at 250°C with strain rate 1.10⁻⁴.s⁻¹. This temperature on the base of previous measurement was selected from the reason of best plasticity.





b)

The results of this testing were following:

Tensile strength $R_m = 35$ MPa and percentage elongation was 250%. Percentage reduction from the reason impossibility of correct fracture area measurement was approximately determined 400%.

Shape of this sample after tensile test describe above in shown in Fig. 3.

Evaluation of microstructure on light microscope NEOPHOT 2 and character of fracture electron microscope JEOL 50A was performed under samples for relevant temperatures.

In Fig. 4a is showed microstructure of alloy in initial cast state. This ones is formed by crystals of matrix on the basis of solid solution of aluminium in magnesium, surrounded by minority phases of the type $Mg_{17}Al_{12}$, or possibly $Mg_{17}(Al, Zn)_{12}$.

Microstructure has distinctly dendritic character; massive phases form almost continuous formations in interdendritic areas, which represent places of initiation and propagation of failure at tensile test. After heat treatment intermetallic phase is almost dissolved as it is seem in Fig. 4b. and the grain boundary shows serration character.

Microstructure near of fracture surface after testing at selected elevated temperatures in the cutting plane of the sample parallel with its axis for alloy in initial as cast state is shown in Figs. 5-8.



Fig. 1. Temperature dependence of mechanical properties alloy AZ91as cast state: a), b)

Fig. 2. Temperature dependence of mechanical properties alloy AZ91 after heat treatment: a), b)



Fig. 3. Shape of ECAP sample after tensile test

At temperatures from 100°C-460°C the partial dissolution of dispersion precipitate are occurred, and at temperatures above 300°C there occurs even coagulation and partial dissolution of the massive phase. These processes are accompanied by forming of micro-pores in interdendritic areas contributing also to initiation of crack propagation along the phase boundary.

The fracture line runs mostly along diminished dendrite boundaries and sub-surface cracks are located in the same areas. As we can see from that figures the maximum of plastic properties at temperatures $(250^{\circ}\text{C}-300^{\circ}\text{C})$ can be connected, among others, with appearing a significant dissolution of dispersion precipitate, decline of strength (and plastic) values at temperature of 460°C is related evidently mainly with formation of continuous non-integrities at the places of massive phase and partly growth of matrix grain (Fig. 8).

Microstructure near of fracture surface after testing at selected elevated temperatures in the cutting plane of the sample parallel with its axis of alloy after heat treatment is shown in Figs. 9-11.

At temperature 100° C a slip bands can be occurred which in temperature interval $200-300^{\circ}$ C disappear and at temperature 400° C a recrystalization process we can see.

Interdendritic character of failure for f alloy in initial as cast state was demonstrated on fracture surfaces at all temperatures. Fracture areas at selected temperatures are shown in Figs. 12-15.

In Figs. 16-18, fracture surfaces of alloy after heat treatment in temperature interval 200-400^oC are shown.

In this case of samples to the change of the character of fracture area became. At lower temperatures the transcrystalline plastic character of fracture vas observed while at temperature brittle fracture has indicated.

The microstructure of sample after rolling before ECAP method application is shown in Fig. 19 and microstructure of samples after ECAP method application is shown in Fig. 20. As shown these figures fine grain microstructure vas occurred.



Fig. 4. Microstructure of used samples: a) as cast state A, b) after heat treatment A1



Fig. 5. Microstructure of samples A after tensile test at 100°C: a), b)

a)

182) Research paper)

Properties



Fig. 6. Microstructure of samples A after tensile test at 250°C: a), b)



Fig. 7. Microstructure of samples A after tensile test at 300°C: a), b)



Fig. 8. Microstructure of samples A after tensile test at 460°C: a), b)



Fig. 9. Microstructure of samples A1 after tensile test at 100°C: a), b)



Fig. 10. Microstructure of samples A1 after tensile test at 200°C



Fig. 11. Microstructure of samples A1 after tensile test at 400°C: a), b)



Fig. 12. Analysis of fracture areas of samples A at 100° C with use of SEM (the same samples as in the Fig. 5)



Fig. 13. Analysis of fracture areas of samples A at 250°C with use of SEM (the same samples as in the Fig. 6)



Fig. 14. Analysis of fracture areas of samples A at 300°C with use of SEM (the same samples as in the Fig. 7)



Fig. 15. Analysis of fracture areas of samples A at 100° C with use of SEM (the same samples as in the Fig. 8)



Fig. 16. Analysis of fracture areas of samples A1 at 100°C withuse of SEM (the same samples as in the Fig. 9)



Fig. 17. Analysis of fracture areas of samples A1 at 200°C withuse of SEM (the same samples as in the Fig. 10)



Fig. 18. Analysis of fracture areas of samples A1 at 400°C withuse of SEM (the same samples as in the Fig. 11)



Fig. 19. Microstructure of samples A1 after rolling



Fig. 20. Microstructure of samples A1 after ECAP method application

4.Conclusions

In this article the mechanical properties, structural and fracture characteristics of the magnesium alloy AZ 91 at elevated temperatures were evaluate.

- At first the microstructure of the alloy in initial cast state is formed by solid solution and by minority phases Mg₁₇(Al,Zn)₁₂ in massive and dispersion form.
- Occurred microstructure has dendritic character, minority phases are comparatively continuously distributed in interdendritic areas, which represent suitable places for initiation and propagation of cracks under load.
- Heating implicated at chosen temperatures of testing alloy in initial cast state causes partial dissolution of minority phases. Homogenisation of microstructure is, however, accompanied by simultaneous forming of inter-granular non-integrities, which is unfavourable from the viewpoint of strength and plastic properties, especially at higher temperatures.
- These structural changes can be connected with increasing of values of tensile strength with increasing test temperatures, as well as with observed changes of plastic properties in the mentioned temperature interval.
- For samples of alloy as cast state during increasing of plastic properties in the temperature interval from 250 to 300^oC were mentioned that some role is played, among others, also by certain homogenisation of microstructure, their decrease at the temperature above 300^oC can be connected with formation of continuous non-integrities, or with melting of residues (of eutectic) phase in interdendritic areas.
- A fracture area in these cases occurs practically at all temperatures basically by inter-crystalline splitting along the boundaries of original dendrites.
- Trans-crystalline plastic character of fracture in small areas at 300°C occurred.
- During heat treatment intermetallic phase is almost dissolved as it is seem in Fig. 4b. and the grain boundary shows serration character.
- Similar temperature dependence of mechanical properties of alloy after heat treatment was observed, but mechanical properties reach more high values.
- In this case of samples of alloy after heat treatment the maximum of plastic properties in the temperature interval from 200 to 250°C is occurred.
- After testing at the temperature above 300⁰C formation of continuous non-integrities is not presented in so much range as in case of alloy as cast state.
- In microstructure pictures at lower temperatures of testing a slip bands appear (Fig. 8, 9) and at temperature testing 400⁰C the recrystalization areas occurred.
- In case of samples of alloy after heat treatment in a part of fracture areas the transcrystalline character of fracture vas observed.
- The possibility of superplasticity effect rise was confirmed.

Acknowledgements

The work was financed by aim of GAČR of the Czech Republic GA-106/04/1346 and sponzored by Project MSM 6198910015, MSM 6198910013, international projects Visegrad S-040-2004, CEEPUS PL-13. Moreover this scientific work is fragmentary financed within the framework of scientific financial resources in the period 2007-2008 as a research and development project R15 0702 headed by Prof. L.A. Dobrzański.

References

- M. Avedesian, H. Baker, ASM Specialty Handbook-Magnesium and Magnesium Alloys, ASM International, USA (1999) 3-84.
- [2] L. Čížek, M. Greger, L. Pavlica, L.A. Dobrzański, T. Tański, Study of selected properties of magnesium alloy AZ91 after heat treatment and forming, Journal of Materials Processing Technology 157-158 (2004) 466-471.
- [3] L. Čížek, M. Greger, L.A. Dobrzański, I. Juřička, R. Kocich, L. Pwlica, T. Tański, Mechanical properties of magnesium alloy AZ91 at elevated temperatures, Journal of Achievements in Materials and Manufacturing Engineering 18 (2006) 203-206.
- [4] L. Čížek, R. Kořený, I. Juřička, J. Maisner, M. Greger, S. Lasek, P. Beťáková, A. Hernas, Structure and Properties of the Selected magnesium Alloys, Proceedings of the 10th International Scientific Conference "Achievements in Mechanical and Materials Engineering" AMME'2001, Gliwice–Zakopane, 2001, 75-78.
- [5] K.A. Dahle, Z.C. Lee, M.D. Nave, P.L. Schaffer, D.H. Johnson, Development of the as-cast microstructure in magnesium-aluminium alloys, Journal of Light Metals 1 (2001) 61-73.
- [6] J.R. Davis, Metals Handbook, Desk Edition, Second Edition, ASM International, The Materials Information Society, USA, 1998.
- [7] L.A. Dobrzański, T. Tański, L. Čížek, Influence of heat treatment on structure and mechanical property of the casting magnesium alloys, Proceedings of the 11th International Scientific Conference "Contemporary Achievements"

in Mechanics, Manufacturing and Materials Science" CAM3S'2005, Gliwice–Zakopane, 2005, (CD-ROM).

- [8] L.A. Dobrzański, T. Tański, L. Čížek, Influence of Al addition on microstructure of die casting magnesium alloys, Journal of Achievements in Materials and Manufacturing Engineering 19/2 (2006) 49-55.
- [9] A. Fajkiel, P. Dudek, G. Sęk-Sas, Foundry engineering XXI c. Directions of metallurgy development and light alloys casting, Publishers Institute of Foundry Engineering, Cracow, 2002.
- [10] H. Friedrich, S. Schumann, Research for a 'New age of magnesium in the automotive industry', Journal of Materials Processing Technology 117 (2001) 276-281.
- [11] M. Greger, L. Čížek, M. Widomska, Structural characteristics Magnesium Alloys along of the Equal Channel Angular Pressing, Proceedings of the International Conference "Advanced Metallic Materials and their Joining", Bratislava, 2004, 55-58.
- [12] M. Greger, R. Kocich, L. Čížek, L.A. Dobrzański, I. Juřička, Possibilities of mechanical properties and microstructure improvement of magnesium alloys, Archives of Materials Science and Engineering 28/2 (2007) 83-90.
- [13] A. Kiełbus, Structure and mechanical properties of casting MSR-B magnesium alloy, Journal of Achievements in Materials and Manufacturing Engineering 18 (2006) 131-134.
- [14] A. Kiełbus, T. Rzychoń, R. Cibis, Microstructure of AM50 die casting magnesium alloy, Journal of Achievements in Materials and Manufacturing Engineering 18 (2006) 135-138.
- [15] X. Ming-Xu, Z. Hong-Xing, Y. Sen, L. Jian-Guo, Recrystallization of preformed AZ91D magnesium alloys in the semisolid state, Materials and Design 26 (2005) 343-349.
- [16] B.L. Mordike, T. Ebert, Magnesium. Propertiesapplications-potential, Journal of Materials Processing Technology 117 (2001) 37-45.
- [17] P. Palček, P. Lukáč, M. Chalupová, Z. Trojanová, Influence of temperature on fracture characteristics selected magnesium alloys, Materials Engineering 3 (2005) 7-10 (in Slovak).
- [18] L. Ptáček, Magnesium alloys-present state of development and exploitation, Proceedings of the Conference METAL 2001, Ostrava - Hradec n. Moravicí, 2001, 1-12 (in Czech).
- [19] T. Tański, L.A. Dobrzański, L. Čížek, Influence of heat treatment on structure and properties of the cast magnesium alloys, Journal of Advanced Materials Research 15-17 (2007) 491-496.