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Vpliv dodatkov antimona (Sb) na mikrostrukturo in mehanske lastnosti zlitine AZ91

Effect of Antimony Additions on Microstructure and Mechanical Properties of AZ91 Alloy

Izvleček

Obdelan je bil učinek dodatka antimona (0,2, 0,5 in 1,0 mas. %) na mikrostrukturo in mehanske lastnosti zlitine AZ91. Dodatek antimona udrobni mikrostrukturo v ulitem stanju in tudi nastale intermetalne delce Mg3Sb2. Ugotovljeno je bilo, da se trdnostne lastnosti z dodanim Sb povečajo, a duktilnost rahlo zmanjša. Najvišje trdnostne lastnosti so bile dosežene pri dodatku 0,5 mas. % Sb.

Ključne besede: magnezij, zlitina AZ91, mikrostruktura, mehanske lastnosti

Abstract

In this study, effect of antimony addition (0.2, 0.5 and 1 wt.% Sb mass fraction) on the microstructure and mechanical properties of AZ91alloy has been studied. Addition of Sb resulted in there finement of the as-cast structure and also the formation of Mg3Sb2 intermetallic particles. The strength properties are found to increase in Sb added alloys with slight reduction in ductility. However, the maximum strength properties are obtained with 0.5 % Sb addition.

Key words: Magnesium, AZ91 alloy, Microstructure, Mechanical properties

1. Uvod

Kot najlažji kovinski konstrukcijski material z veliko specifično trdnostjo, specifično togostjo in dobro strojno obdelovalnostjo se magnezij in njegove zlitine zaradi svoje majhne gostote vse bolj uporabljajo na mnogih področjih tehnike, vključno s prenosnimi mikroelektronskimi napravami, telekomunikacijami, letalsko in vesoljsko tehniko in avtomobilsko industrijo [1]. Posebno zlitina AZ91 (MgAl9Zn0,8Mn0,2) je najbolj uporabljana magnezijeva zlitina. Njena uporaba predstavlja okoli 90 % celotne porabe vseh ulitih magnezijevih izdelkov [2]. Analizirali smo vpliv dodatkov stranskih zlitinskih elementov, kot so Ca, Bi, Pb in redke zemlje, na izboljšavo litja,

1. Introduction

As the lightest metallic structural material, high specific strength, specific stiffness and machinability, magnesium and its alloys are increasingly used in many engineering areas including portable microelectronics, telecommunication, aerospace and automobile industries due to their low density [1]. Especially, alloy AZ91 (Mg-9Al-0.8Zn-0.2Mn) is the most favoured magnesium alloy, being used in approximately 90 % of all magnesium cast products [2]. Additions of minor alloying elements such as Ca, Bi, Pb and rare earth (RE) to AZ91alloy have been studied to improve casting, microstructure stability or creep properties of the alloy [3-8].

mikrostrukturno stabilnost ali lastnosti, povezane z lezenjem zlitine [3-8].

Izboljšanje nateznih lastnosti magnezijevih zlitin vrste AZ, tako pri sobni kot pri povišanih temperaturah, se lahko doseže z uporabo stranskih zlitinskih elementov, kot je Sb, ki tvori stabilne nove faze v magnezijevih zlitinah v ulitem stanju. Glede na binarni fazni diagram Mg-Sb [9] je topnost antimona zanemarljiva, zato se pričakuje nastanek zelo stabilne intermetalne faze Mg3Sb2. V literaturi je več podatkov, da se mehanske lastnosti magnezijevih zlitih lahko izboljšajo z dodajanjem Sb [10,11]. Nayyeri in Mahmudi [10] sta npr. raziskovala odpornost proti lezenju ulite binarne zlitine Mg5Sn z dodanim Sb. Alizadeh in Mahmudi [11] sta analizirala vpliv dodatka Sb na mikrostrukturo in visokotemperaturne mehanske lastnosti zlitine Mg-Zn v ulitem stanju. Literaturni pregled je odkril, da je učinek Sb kot stranskega zlitinskega elementa na magnezijeve zlitine vrste AZ, posebno na AZ91, v ulitem stanju majhen. Zato je bil cilj te raziskave ugotoviti učinek manjših dodatkov Sb na mikrostrukturo in mehanske lastnosti zlitin AZ91 v ulitem stanju.

2. Poskusi

Bloke Mg, Al, Zn in Sb z najmanjšo čistostjo 99,9 % smo nabavili pri Skarya Metal Co., Turčija. Zlitine smo izdelovali tako, da smo skupaj talili čisti Mg in dodatke Al v grafitnem loncu pod zaščitno atmosfero argona pri 750 oC ter potem pred litjem počakali 20 min. Zn in Sb smo dodali 5 min pred litjem, da bi se izognili izgubam Zn in Sb zaradi izparevanja. Izbrali smo deleže Sb 0,2 mas. %, 0,5 mas. % in 1,0 mas. %. Kemične sestave analiziranih zlitin so prikazane v razpredelnici 1.

Enhancement in the tensile properties of AZ series magnesium alloys, both at ambient and elevated temperatures, could be facilitated by using minor alloying elements such as Sb, which produce stable second phase constituent in the as-cast condition of magnesium alloys. According to the Mg-Sb binary phase diagram [9], the solubility of Sb is negligible, therefore, highly stable intermetallic phase Mg3Sb2 is expected to form. There are several reports in the literature that the mechanical properties of magnesium alloys could be increased by Sb [10,11] addition. For example, Nayyeri and Mahmudi [10] studied creep resistance of a Sb added cast Mg-5Sn binary alloy. Alizadeh and Mahmudi [11] studied the effect of Sb addition on the microstructure and high-temperature mechanical properties of as-cast Mg-Zn alloy. Literature review revealed that the effect of Sb (as a minor alloying addition) to AZ series Mg alloys, especially AZ91 alloy in as cast condition, is scarce. Therefore, the objective of the present study was to characterize effect of minor Sb additions on the microstructure and mechanical properties of as-cast AZ91 alloys.

2 Experimental procedure

Mg, Al, Zn, Sb ingots with a minimum purity of 99.9 % were purchased from Sakarya Metal Co., Turkey. The alloys were prepared by melting pure Mg together with Al alloying additions in a graphite crucible under Ar gas atmosphere at 750 oC and then held for 20 min before pouring. Zn and Sb additions were carried out 5 min before casting to avoid losses of Zn and Sb due to vaporization. The amount of Sb content in AZ91 alloy has been selected as 0.2, 0.5 and 1.0 %. The chemical compositions of the studied alloys are shown in Table 1.

Razpredelnica 1. Kemična sestava zlitin

Table 1.Chemical composition of the alloys

Zlitina / Alloy	Elementi (mas.%) / Elements(%, mass fraction)				
	Al	Zn	Mn	Sb	Mg
AZ91	9.23	0.96	0.17	-	ost. / Bal.
AZ91+0.2Sb	8.89	1.08	0.21	0.19	ost. / Bal.
AZ91+0.5Sb	9.12	0.98	0.23	0.53	ost. / Bal.
AZ91+1Sb	8.71	1.03	0.19	0.96	ost. / Bal.

Staljene zlitine smo ulili pod zaščitno atmosfero SF6 v predgreto (250 oC) litoželezno kokilo. Za preiskave smo uporabili preskušance v ulitem stanju. Trdoto smo ugotavljali z merilnikom trdote po Vickersu z obremenitvijo 50 N. Preskušance za natezne preskuse, dolge 40 mm in s premerom 8 mm, smo izdelali s strojno obdelavo. Natezni preskusi so potekali v skladu s standardom ASTM E 8M-99 s hitrostjo obremenjevanja 0,5 mm/min pri sobni temperaturi. Za mikrostrukturno analizo so bili pripravljeni kockasti vzorci 10 mm x 10 mm x 10 mm, izrezani iz sredine vsakega preskušanca. Eno ploskev smo zbrusili na brusilnih papirjih 220, 400 in 600, nato polirali z diamantno pasto (1 μm). Za mikrostrukturne preiskave smo uporabili svetlobni mikroskop in vrstični mikroskop Phillips XL30 ESEM.

3. Rezultati in razprava

3.1 Mikrostruktura

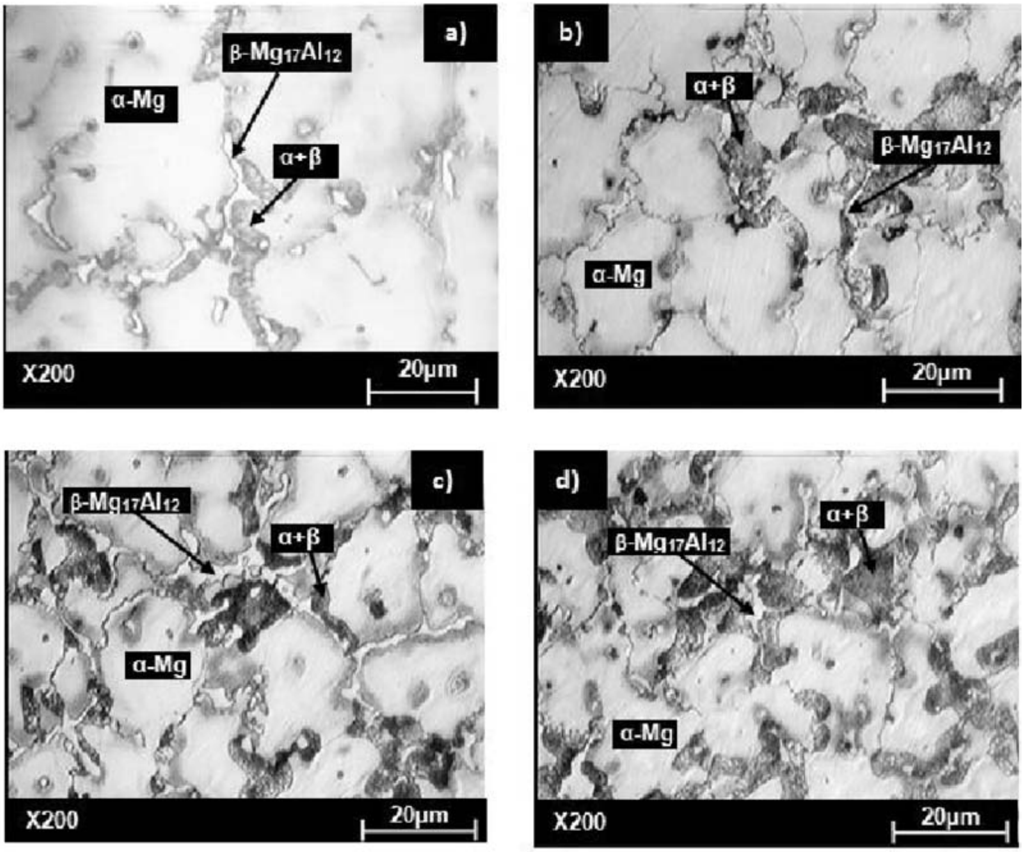
Slika 1 kaže mikrostrukture zlitin AZ91 + xSb (x = 0, 0,2, 0,5, 1,0) v ulitem stanju. Kot se vidi iz slike 1(a), je zlitina AZ91 v ulitem stanju sestavljena iz primarne α-Mg osnove, popačenega evtektika β-Mg17Al12 in sekundarno izločene faze

The molten alloys were cast under protective SF6 gas into a preheated (250 oC) cast iron mould. The alloy specimens were used in as-cast condition for the experiments. The hardness values were determined by Vickers hardness testing with a load of 50 N. The tensile test samples having 40 mm in length and 8 mm in diameter were machined. The tensile tests were performed (ASTM E 8M-99) with a crosshead speed of 0.5mm/min at room temperature. Samples having 10x10x10 mm cube were cut from the centre of the each specimen then one face of the cube subsequently ground with 220, 400 and 600 grit emery papers followed by polishing with 1μm diamond paste for microstructural evaluations. Microstructural evaluations were carried out by optical light microscopy and Phillips XL30 ESEM scanning electron microscopy (SEM).

3. Results and discussion

3.1 Microstructure

Figure 1 shows the microstructures of as-cast AZ91 + xSb (x = 0, 0.2, 0.5, 1.0 %) alloys. As shown in Fig.1(a), as-cast AZ91 alloy consists of primary α-Mg matrix, divorced eutectic β-Mg17Al12 phase and



Slika 1. Mikrostrukture zlitin AZ91(a), Z91 + 0,2% Sb (b), AZ91 + 0,5% Sb (c) in AZ91 + 1% Sb (d) v svetlobnem mikroskopu

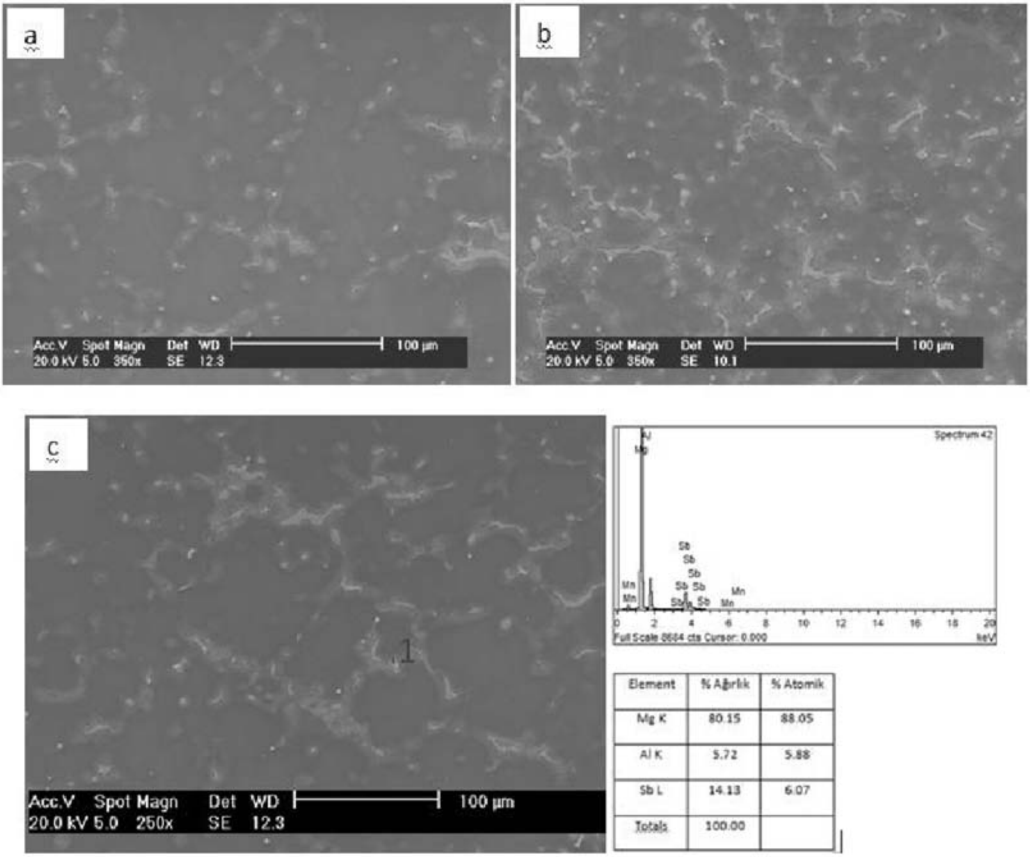
Figure 1. Optical microstructures of AZ91(a), Z91+0.2%Sb (b), AZ91+0.5%Sb (c) and AZ91+1%Sb (d) alloys

β-Mg17Al12 . Izločki evtektične faze tvorijo mreže na mejah zrn. Z dodatkom Sb se groba evtektična faza udrobni in postane nezvezna, a pojavijo se nove kosmičaste in zrnate faze.

Slika 2(a)–(c) kaže SEM-posnetke zlitine AZ91 z dodatki Sb in brez njih pri večji povečavi. Dobro se vidi, da dodatek Sb zlitini AZ91 pretvori lamelaste evtektike v popolnoma popačene β evtektike. Že prej so poročali [12-14], da je nastanek popolnoma popačenega, deloma popačenega in

secondary precipitated β-Mg17Al12 phase. The eutectic phase precipitates in the form of network at grain boundaries. With the addition of Sb, coarse eutectic phase is refined and becomes discontinuous, and new flaky and granular phases occur.

Fig. 2(a)-(c) show higher magnification of SEM micrographs of AZ91 alloy with and without Sb added alloys. Evidently, addition of Sb to AZ91 alloy transformed lamellar eutectics into fully divorced β eutectics. It has been previously reported [12-14] that



Element	% Ağırlık	% Atomik
Mg K	80.15	88.05
Al K	5.72	5.88
Sb L	14.13	6.07
Totals	100.00	

Slika 2. SEM-posnetki zlitin AZ91 + 0,2 % Sb (a), AZ91 + 0,5 % Sb (b) in AZ91 + 1 % Sb (c) pri večji povečavi

Figure 2. Higher magnification of SEM micrographs of AZ91+0.2%Sb (a),AZ91+0.5%Sb (b) andAZ91+1%Sb (c) alloys

lamelastega evtektika, ki nastajajo v magnezijevih zlitinah, odvisen od dodatkov zlitinskih elementov.

Kot kaže slika 2c se je nova faza pojavila kot popolnoma popačeni β-evtektik pri zlitini z 1,0 mas. % Sb. EDS-analiza je pokazala, da ta faza vsebuje Sb kot tudi Mg in Al v fazi Mg3Sb2. Prisotnost faze Mg3Sb2 v zlitini AZ91, ki vsebuje Sb, je očitna, kot kaže slika 2c.

fully divorced, partially divorced and lamellar eutectic morphologies can be formed in magnesium alloys depending on alloying additions.

A different phase in the fully divorced β eutectics have been appeared in 1.0 % Sb containing alloys as shown in Fig. 2(c). EDS analysis showed that this phase contains Sb as well as Mg and Al being Mg3Sb2 phase. Presence of Mg3Sb2 phase in Sb containing AZ91 alloy is evident as shown in Fig. 2(c).

3.2 Mehanske lastnosti

Kot kaže slika 3, se trdota HV veča z dodajanjem Sb. Slika 3 tudi kaže, da natezna trdnost pri pretrgu in meja tečenja dosežeta največjo vrednost pri deležu Sb 0,5 mas %. Na drugi strani večji delež Sb (> 0,5 mas. %) zmanjša natezno trdnost pri pretrgu in napetost tečenja. To je lahko zato, ker postanejo izločki faze Mg3Sb2 bolj grobi. Poleg tega smo v β-evtektikih opazili nove delce, ki vsebujejo Sb (Mg3Sb2) ter razpoke med β-evtektiki in Mg3Sb2, kot jasno kaže slika 2c. Zato prekomerno dodajanje Sb vodi do bolj grobih delcev Mg3Sb2 in povečanega pojavljanja razpok v vročem, o čemer smo razpravljali že prej, in to je vzrok za zmanjšanje trdnosti in plastičnost, kar je v popolnem soglasju z delom Jihue in sodelavcev [15]. Povečanje trdote HV, natezne trdnosti pri pretrgu in napetosti tečenja so pripisali udrobnjenju mikrostrukture. Odnos med velikostjo zrn in napetostjo tečenja se lahko izrazi z Hall-Petschovim obrazcem:

$$\sigma_s = \sigma_o + kD^{-1/2} \qquad 1)$$

kjer je σ_s napetost tečenja, σ_o in k sta konstanti in D je velikost zrn. Iz enačbe (1) se vidi, da se napetost tečenja veča z zmanjševanjem velikosti zrna. In zmanjševanje velikost zrn pomeni povečanje mej zrn, kar lahko izboljša mehanske lastnosti magnezijeve zlitine, ker kristalne meje zavirajo gibanje dislokacij.

4 Sklepi

Naslednji sklepi se lahko naredijo iz te raziskave:

- pri dodatku 0,2 mas. % Sb je bila mikrostruktura drobnejša kot pri osnovni zlitini AZ91. Ko se je delež Sb povečal

3.2 Mechanical Properties

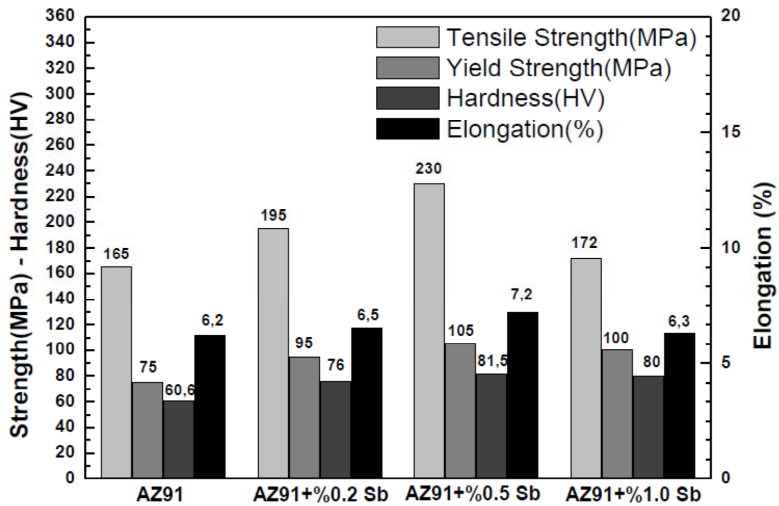
It can be seen that the HV increases as the Sb content of the AZ91 alloy increases investigated as depicted in Fig. 3. Fig. 3 also shows that the UTS and YS reach the peak values as Sn content is 0.5 %. On the other hand, higher Sb content (>0.5 %) also had resulted in the reduction of a UTS and YS. This was believed to be due to coarsening effect of Mg3Sb2 precipitates. Indeed, a different particle containing Sb (Mg3Sb2) in the β eutectics has been observed and crack at the interface between β eutectics and Mg3Sb2 is evident as shown in Fig. 2(c). Therefore, excessive Sb addition leads to the coarsening of Mg3Sb2 particles as well as increased hot tearing as discussed earlier, and thus results in the decline of strength and plasticity of the alloy entirely consistent with the work of Jihua et al. [15].The increase in HV, UTS and YS was attributed to decrease in grain size of the microstructure. The relationship between grain size and YS can be expressed according to Hall-Petch formulation as follows;

$$\sigma_s = \sigma_o + kD^{-1/2} \qquad 1)$$

where σ_s is the YS, σ_o and k are constant, and D is the grain size. It is known from Eq.(1) that the YS increases with decreasing grain size. And decreasing the grain size means increasing grain boundaries, which can improve the mechanical properties of magnesium alloy because the grain boundaries can hinder the dislocation motion.

4 Conclusions

The following conclusions can be drawn from the present study:



Slika 3. Natezna trdnost pri pretrgu (UTS), trdota HV, raztezek (EL) in napetost tečenja (YS) zlitine AZ91 kot funkcija deleža Sb

Figure. 3. Ultimate tensile strength (UTS), hardness (HV), elongation (EL) and yield strength (YS) of AZ91 alloy as a function of its Sb content

nad 0,5 mas. %, so zrna začela rasti;

- z dodanjem Sb se je v mikrostrukturi zlitine AZ91 pojavila intermetalna faza Mg3Sb2. Delež intermetalne faze Mg3Sb2 se je večal z večanjem deleža Sb v zlitini;
- natezna napetost pri pretrgu, napetost tečenja in trdota HV so se znatno povečale z dodajanjem Sb do 0,5 mas. %, nato so se do dodatka 1,0 mas. % začele manjšati.

- The microstructure was refined and grain size was reduced with 0.5 % Sb addition to base AZ91 alloy. As Sb addition is increased above 0.5 %, the grains begin to coarsen
- Mg3Sb2 intermetallic phase has appeared in the microstructure of AZ91 alloy with Sb addition. The amount of the Mg3Sb2phases increased with increasing the Sb content of the alloys.
- UTS, YS and HV were increased considerably with addition of up to 0.5 % Sb then decreased with increasing Sb content up to 1.0 %.

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