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Effect of Co-addition of Sn, Traces of RE and Ca on the Mechanical and Micro Structural Properties of As-cast AZ80 Alloy

Madhusudhanprasad MANCHALA *, **Ramachandra Raju VEGESNA**

Department of Mechanical Engineering, JNT University College of Engineering Kakinada (A), Kakinada, Andhra Pradesh, 533003, India

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In this study, the effect of co-addition of tin (0, 6 and 9 wt.%), cerium (0, 0.3 and 0.6 wt.%), gadolinium (0, 0.6 and 0.9 wt.%) and calcium (0, 0.6 and 0.9 wt.%) on the mechanical and microstructural properties of AZ80 magnesium alloy is investigated. The results indicate that the mechanical properties are improved for all the as-cast alloys when compared to base alloy except for one alloy which is due to the presence of Mg-Sn-Ca phase in high volume fraction. Also, it is found that the existence of Al_2Ca, Mg_2Ca, Al_2RE, MgSnRE and Mg_2Sn phases have a significant impact on the mechanical and microstructural characteristics of the as-cast AZ80alloys. It is observed that without the addition of Sn content, the best room temperature tensile properties are obtained for the alloy containing 1.5 wt.% of RE and 0.9 wt.% of Ca. With the addition of Sn, it is found that the best room temperature tensile properties are obtained for the alloy containing 6 wt.% of Sn, 0.9 wt.% of RE and 0.5 wt.% of Ca.

Key words: magnesium alloy, rare earth metal, casting, microstructure, mechanical properties.

1. INTRODUCTION

Due to their lower density, excellent damping capacity, and widespread availability, magnesium (Mg) alloys are being used [1 – 3] in areas that are experiencing significant growth, such as the automotive and aerospace industries. Generally, for low-temperature applications, Mg cast alloys like Mg-(6 to 9 wt.%) aluminum (Al) alloys are frequently used. Furthermore, these Mg-Al alloys can be used for high temperature applications when they are alloyed with proper alloying elements. The results from several reports [4 – 7] demonstrate how the inclusion of Tin (Sn) improved the mechanical properties and corrosion rates of the Al series of magnesium alloys. However, this improvement is limited due to the formation of the $Mg_{17}Al_{12}$ phase. Due to the ability to enhance creep resistance and tensile strength, calcium (Ca) is generally added to Mg alloys [8]. Yanfu Chai, et al studied [9] the properties of Mg-Sn-Zn alloy with the minor addition of Ca content and found that the mechanical properties are improved while the Mg_2Sn phase transforms into Ca-Mg-Sn and Mg_2Ca phases. Also, the combination of Sn and Ca in Mg-Zn-Al alloy [10] is found to enhance the mechanical properties. Recently, investigations have been carried out [11] on the addition of rare earth (RE) elements in Mg alloys to improve the mechanical properties and refine microstructure. It is reported that [12 – 14] alloying of either single RE or a combination of REs to the AZ series alloy led to the grain refinement and Al_2REphase formation which in turn improved the corrosion resistance and mechanical properties. Jiang, et al pointed out [14] that when Neodymium (Nd) and Gadolinium (Gd) are added to AZ80 alloy in small quantities, an effective modification in the microstructure is found to result in the growth of Al-RE-Mn, $Al_{11}RE_3$, and Al_2RE phase. Yang, et al studied [15] the variation of microstructure and age hardening behaviour in Mg-6Zn-1Mn-4Sn alloy with minor Gd additions and found the finely grown Mg_7Zn_3, Mg_2Sn, and Mg-Sn-Gd phases. They pointed out that due to the refinement of grains, higher density precipitation occurs, and the retainment of dislocations resulted in the increase of strength in 0.2 wt.% Gd alloy.

Cerium (Ce) is one of the abundantly available RE which can significantly affect the properties of Mg alloys when added in small amounts. It is reported [16] that 0.2 wt.%Ce in Pure Mg followed by its extrusion resulted for improved ductility. When Ce is added up to 1.5 wt.% in ZK60 alloy, it is found to result [17] in the reduction of average grain size and improvement in ultimate tensile strength but at the cost of ductility.

Furthermore, Chaojie Che, et. al [18] investigated the effect of the co-addition of Si, Ca & RE on AZ91 alloy and found that these alloying elements influenced the microstructure and mechanical properties. Similarly, other studies [19 – 21] also reported that trace addition of alloying elements leads to the improvement of properties and change of microstructure.

Although from the above studies, it is evident that combined addition or trace additions of alloying elements can improve the mechanical properties in certain Mg alloys, the combined effect of alloying elements on Mg-8Al alloy is rarely reported. In the present work, the influence of the co-addition of Sn, REs and Ca on Mg-8Alalloy (B)is investigated through the casting route.

2. EXPERIMENTAL DETAILS

To study the combined influence of Sn (0, 6 and 9 wt.%) content and traces of Ce (0, 0.3 and 0.6 wt.%), Gd

*Corresponding author. +91-9966915354.
E-mail: *msprasad@jntucek.ac.in*(M. Manchala)

(0, 0.6and 0.9 wt.%) and Ca (0, 0.5 and 0.9 wt.%) on the properties of AZ80 alloy, nine alloys are chosen (based on the Design of experiments through Taguchi approach) and cast. The chemical compositions of the casted alloy(s) are listed in Table 1. Commercially available pure metals like Mg, Al, Zn, Si, Sn, Gd, and Ce are used. Manganese (Mn) and calcium (Ca) are added as Mg-15%Mn and Mg-10%Ca master alloys. A temperature refractory crucible with pure magnesium ingots is placed inside an INDFURR Induction furnace and heated initially up to 850 °C in the presence of high purity SF_6 gas (99.9 %). All the necessary alloying elements are added to the magnesium melt. The alloy is maintained at a temperature of 720 °C for around 40 minutes. Then, the molten metal is transferred to a preheated (300 °C – 400 °C) alloy steel mould of volume 25 cm × 20 cm × 3 cm. An INDFURR Oven is used for preheating the steel mould. Fig. 1 shows the experimental setup, crucible, steel mould, oven, and as-cast samples.

Casted samples are tested according to ASTM B557-02a standard using an FIE-UTE Universal Testing Machine to find the ultimate tensile strength (UTS), yield strength (YS) and percentage elongation (%E). The hardness test is conducted using a FIE- B3000 Brinell hardness machine with a tungsten (W) indenter. Samples are tested under an optical microscope (OM) after grit by 180, 240, 320, 400, 600, 800, 1000, 1500, and 2000 grits followed by polishing with Al_2O_3 powder and etched with acetic acid- picric acid-ethanol solution. Scanning electron microscope (SEM) and Energy dispersive spectroscopy (EDS)are used to observe the microstructure in detail. X-ray diffraction (XRD) is used to identify the phase constituents of the alloys.

3. RESULTS AND DISCUSSION

In the present work, Sn, Gd, Ce and Ca metals are added to Mg-8Al (B) based magnesium alloy through the casting route. The impact of these elements on the mechanical and micro structural properties of as-casted Mg-8Al based alloys is presented here.

3.1. Microstructural study

XRD patterns exhibited by casted alloys are illustrated in Fig. 2. The base alloy B1111 is found to consist of a primary Mg phase and a secondary Mg17Al12 phase. In general [4, 8, 15, 23], greater electronegativity (EN) between elements will lead to the easier formation of intermetallic compounds during solidification.

Table 1. Chemical composition (wt.%) of as-cast AZ80 (B) alloys

Alloy	Al	Zn	Si	Mn	Sn	Ce	Gd	Ca	Mg
B1111	8.5	0.5	0.1	0.12	–	–	–	–	Bal.
B1222	8.5	0.5	0.1	0.12	–	0.3	0.6	0.5	Bal.
B1333	8.5	0.5	0.1	0.12	–	0.6	0.9	0.9	Bal.
B2213	8.5	0.5	0.1	0.12	6	0.3	–	0.9	Bal.
B2321	8.5	0.5	0.1	0.12	6	0.6	0.6	–	Bal.
B2132	8.5	0.5	0.1	0.12	6	–	0.9	0.5	Bal.
B3312	8.5	0.5	0.1	0.12	9	0.6	–	0.5	Bal.
B3123	8.5	0.5	0.1	0.12	9	–	0.6	0.9	Bal.
B3231	8.5	0.5	0.1	0.12	9	0.3	0.9	–	Bal.

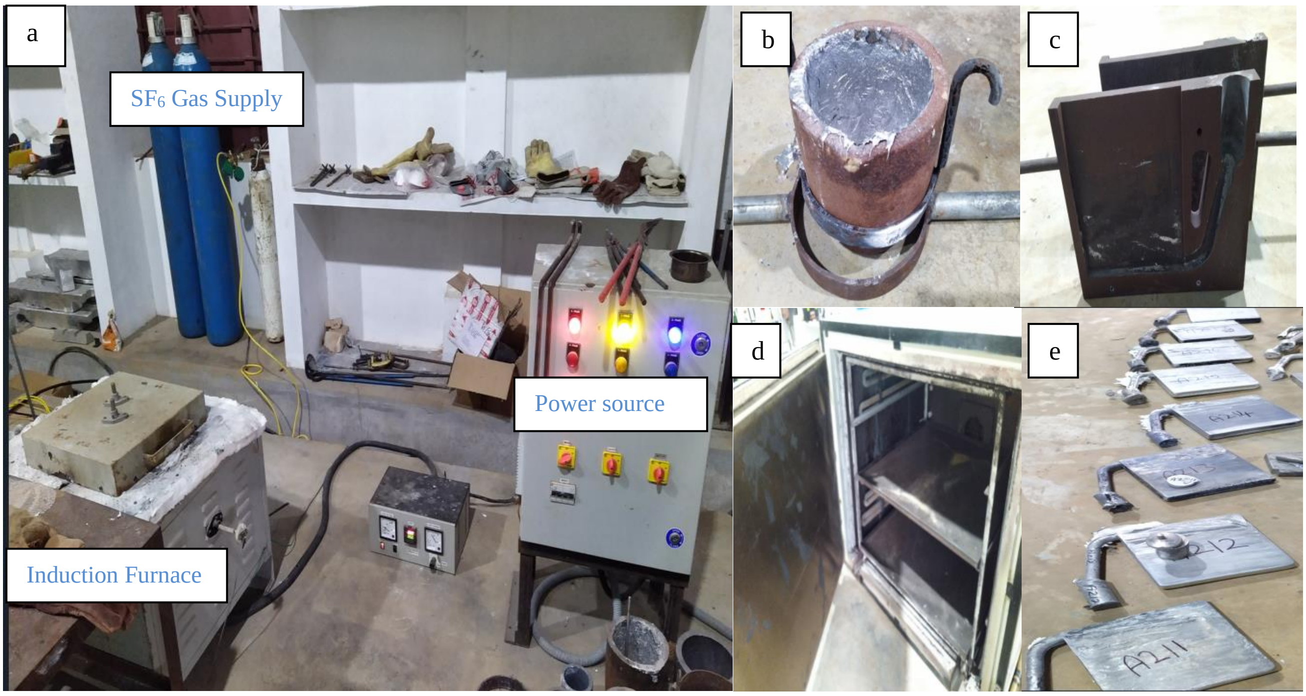


Fig. 1. a – experimental set up; b – refractory crucible; c – steel mould; d – oven; e – as-cast samples (Photo courtesy @ MatRICS, Nagercoil, India)

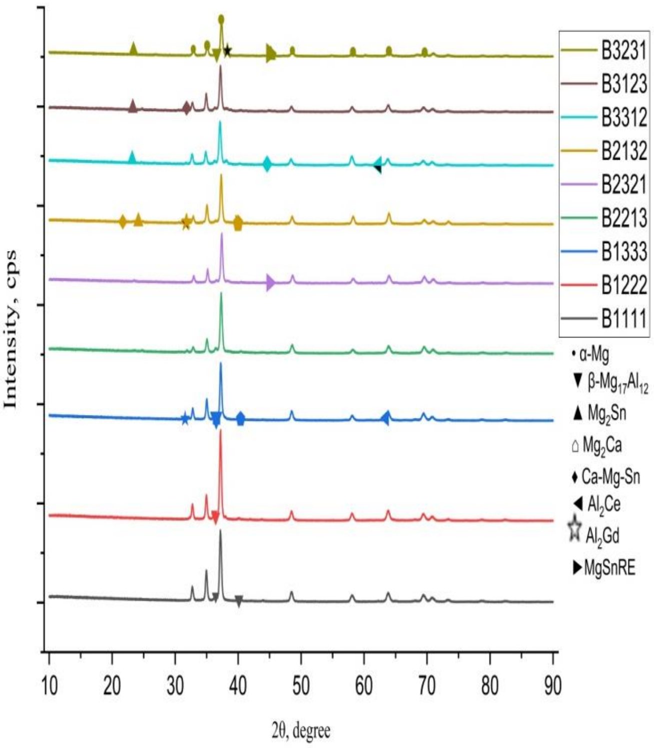


Fig. 2. XRD patterns of casted AZ80 alloys

The electro negativity difference between Mg-Al, Mg-Sn, Mg-Si, Al-Ce, Al-Gd, and Al-Ca are 0.30, 0.65, 0.59, 0.45, 0.41 and 0.61 respectively. As reported by B. Pourbahari, et. al [22], the Al_2RE phase is observed to be formed at the expense of the secondary $Mg_{17}Al_{12}$ phase. It is also observed that when Sn is added to Mg-8Al alloy, the formation of Mg_2Sn (with EN of 0.65) is predominant when compared to Al-Sn (with EN of 0.35). Furthermore, for alloys with RE content (> 0.3 wt.%), Al_2RE is likely to be formed and for the alloys with Ca addition, Al_2Ca formation is easier when compared to Mg_2Ca based on EN. For B2213, B3312 and B3123 alloys, the Mg-Sn-Ca phase is observed to be formed. The above observations can be confirmed from XRD peaks and SEM-EDS analysis.

Optical microscopic images of cast alloys are presented in Fig. 3. In B1111 alloy, secondary $Mg_{17}Al_{12}$ phase is found to be formed which is verified from the XRD pattern. This phase is found to be distributed along the boundaries of grains and into the spaces between dendrites. For B1222 alloy, coarse grains are observed. The formation of a secondary $Mg_{17}Al_{12}$ phase is observed for all the casted alloys. In B1333 alloy, fine grains are formed when compared to B1222 alloy and fine precipitates (small-block shaped) are found [14] to get dispersed along the grain boundaries and within the grains. These fine precipitates can be attributed to Al_2RE and Mg_2(Si, Ca) phases observed from XRD peaks and SEM-EDS analysis. For B2213, B3312 and B3123 alloys more coarsening trend of grains is observed. In B2321, and B2132 alloys, the precipitate formation is more especially in B2132 alloy which is attributed to the presence of small-block shaped Al_2RE and Mg_2Sn phases. In B2213 and B3123 alloys, a needle like Mg-Sn-Ca phase is found [23] to be formed. This formation of Mg-Sn-Ca will reduce the amount of Mg_2Sn which can be ascribed to earlier nucleation of Mg-Sn-Ca phase prior to Mg_2Sn according to EN value [15]. For B3231 alloy, lesser coarsening of grains with small-block shaped Al_2RE and Mg_2Sn phases are seen to be dispersed along the grain borders and within the grains.

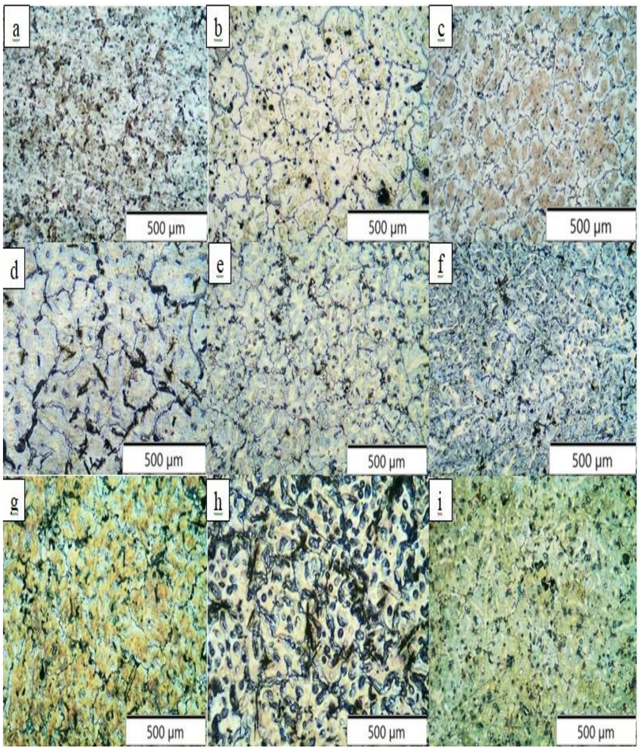


Fig. 3. Optical microscopic images: a – B1111; b – B1222; c – B1333; d – B2213; e – B2321; f – B2132; g – B3312; h – B3123; i – B3231 alloys

3.2. SEM analysis

The SEM image for B1111 as cast alloy is given in Fig. 4 a and EDAX spectrum is shown in Fig. 4 b. The EDAX result for Fig. 4 a is listed in Table 2. The secondary $Mg_{17}Al_{12}$ phase is observed to be dispersed in the primary Mg phase. The small precipitates are found to be formed as Mg_2Si particles. However, the diffraction peak for the same is not visible in the XRD pattern. This similar trend reported elsewhere [14]is due to the grown phase's low volume proportion as compared to primary or secondary phases.

The SEM image for as cast B2132 alloy is displayed in Fig. 5 a and EDAX spectrum is shown in Fig. 5 b. The EDAX result for Fig. 5 a is listed in Table 3. The secondary $Mg_{17}Al_{12}$ phase is observed to be dispersed in the primary phase in this alloy. The growth for Mg_2Sn, Al_2Gd and Mg_2Ca phases is confirmed by the elemental mapping and the XRD peaks. The volume fraction of Mg_2Sn and Al_2Gd is found to be more than Mg_2Ca since the volume proportion of Ca is less. This observation is confirmed by SEM-EDS analysis and the XRD peaks.

The SEM image for as cast B1333 alloy is illustrated in Fig. 6 a and EDAX spectrum is shown in Fig. 6 b. The EDAX result for Fig. 6 a is listed in Table 4. In this alloy, Al_2RE, Mg_2(Si, Ca) phases are observed to exist in precipitate form. It is also observed that the volume proportion of the Al_2Ca phase is more prevalent than that of the Al_2RE and Mg_2(Si, Ca) phases.

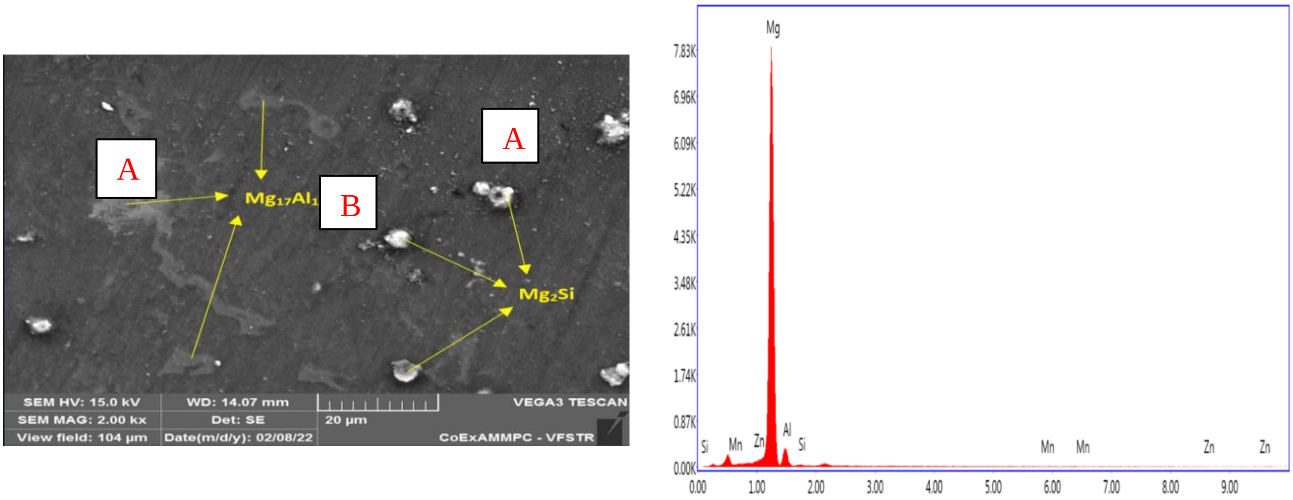


Fig. 4. a – SEM image; b – EDAX spectrum of B1111 alloy

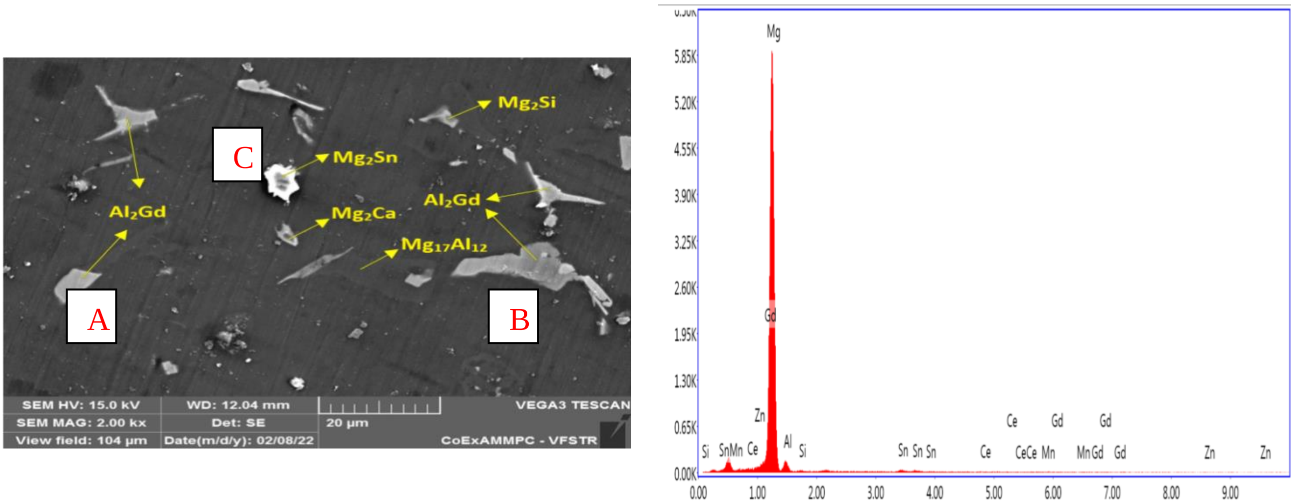


Fig. 5. a – SEM image; b – EDAX Spectrum of B2132 Alloy

Table 2. EDAX results of Fig. 4 a

Region (R)	Mass fraction percentages				
	Mg	Al	Si	Mn	Zn
A	70.73	25.39	0.71	0.90	2.27
B	74.56	3.42	20.76	0.26	1.00

Table 3. EDAX results of Fig. 5 a

R	Mass fraction percentages							
	Mg	Al	Si	Mn	Zn	Sn	Ca	Gd
A	82.2	8.6	0.1	0.6	2.0	4.4	0.9	1.2
B	79.2	2.9	0.3	0.4	0.6	1.0	15.1	0.5
C	68.2	1.0	0.4	0.3	0.5	25.6	3.8	0.2

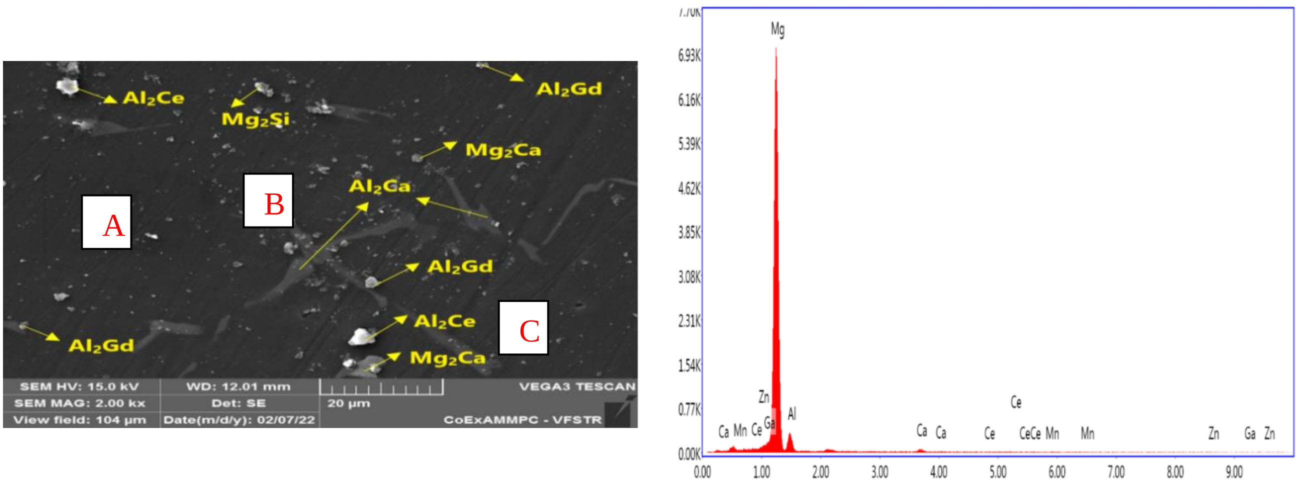
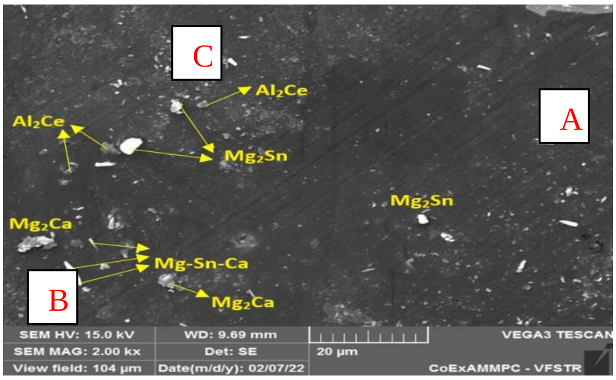
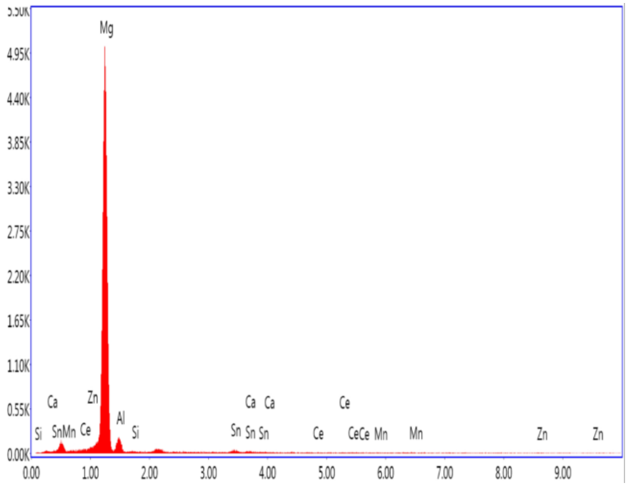


Fig. 6. a – SEM image; b – EDAX Spectrum of B1333Alloy



a



b

Fig. 7. a – SEM image; b – EDAX Spectrum of B2213 Alloy

Table 4. EDAX results of Fig. 6 a

R	Mass fraction percentages						
	Mg	Al	Mn	Zn	Si	Ca	Ce
A	84.7	9.1	0.49	2.72	0.4	1.8	0.9
B	11.0	72.1	0.4	0.5	0.2	15.1	0.7
C	73.2	2.9	0.3	0.4	0.6	21.6	1.0

Table 5. EDAX results of Fig. 7 a

R	Mass fraction percentages							
	Mg	Al	Si	Mn	Zn	Sn	Ca	Ce
A	83.3	7.52	0.4	0.5	4.55	2.87	0.15	0.71
B	22.4	1.5	0.1	0.6	0.9	62.0	11.6	0.9
C	6.4	70.4	0.0	0.1	0.5	1.5	0.9	20.2

The precipitates are observed to be randomly dispersed in both of the primary Mg and secondary $Mg_{17}Al_{12}$ phases.

The SEM image of the B2213 alloy is presented in Fig. 7 a and EDAX spectrum is shown in Fig. 7 b. The EDAX result for Fig. 7 a is listed in Table 5. In this alloy, Mg_2 (Sn, Ca) phase and Al_2Ce phase are found to grow in the form of precipitates. From the OM image of B2213 alloy, it is evident that needle like Mg-Sn-Ca phase is grown as verified form XRD peaks and SEM-EDS image.

3.3. Mechanical properties

The estimated properties like UTS, YS, %E, Brinell hardness number (BHN) of the cast alloys are presented in Table 6, while their trends are represented in Fig. 8. Also, the percentage enhancement of properties when compared to base alloy i.e., B1111 alloy are indicated in Table 6. The as-cast alloy B2132 is found to possess UTS of 208 MPa followed by B1333 with 204 MPa and B2321 with 193 MPa. Likewise, YS is found to be high for B3231 with 155 MPa followed by B2132 with 152 MPa and B2321 with 151 MPa. The percentage elongation is high for B2132 and B1333 at 9 % followed by B2321 with 7.5 %. The hardness is found to be high for B3312 and B3231 with 65 BHN followed by B3123 with 63 BHN. For B2132 alloy, the observed enhanced mechanical properties are attributed to the decrease in volume fraction of $Mg_{17}Al_{12}$ phase due to the prior formation of thermally stable, high melting point temperature Mg_2Sn, Al_2Gdphases and to the presence of low volume fraction of Mg_2(Si,Ca) phase. For B1333 alloy, the enhancement of properties is attributed to relatively finer grain size and the presence of precipitates which lead to dispersion strengthening as reported by Yang Zhao, et. al [15]. Also, the properties of Mg-8Al alloy are observed to be enhanced with the presence of the Al_2Ca phase and fine Al_2RE and Mg_2(Si, Ca) phases by preventing slippage dislocation motion which is well in agreement with previous studies [14, 21]. In B2213 alloy, coarse grains are found to exist with a relatively high-volume fraction of needle like Mg-Sn-Ca phase [23]as observed from OM image and SEM-EDS analysis. As such, they act as a potential site for the formation of micro cracks resulting in decreased properties. This decrement in the properties is also attributed to the presence of coarse precipitates dispersed in both the primary and secondary phases.

Table 6. Mechanical properties of as-cast AZ80alloysand their percentage enhancement at ambient temperature

Cast alloy	UTS, MPa	UTS percentage enhancement	YS, MPa	YS percentage enhancement	Percentage elongation, %E	Elongation percentage enhancement	BHN	Hardness percentage enhancement
B1111	136	–	118	–	1.82	–	52	–
B1222	152	11.8	118	0	6.7	268	56	7.7
B1333	204	50	138	17	9	394	53	2
B2213	127	-6.6	118	0	4.2	131	58	11.5
B2321	193	42	151	28	7.5	312	49	-6
B2132	208	53	152	29	9	394	60	15.4
B3312	151	11	138	17	3.2	76	65	25
B3123	153	12.5	145	23	6.6	263	63	21
B3231	168	23.5	155	31	5.3	191	65	25

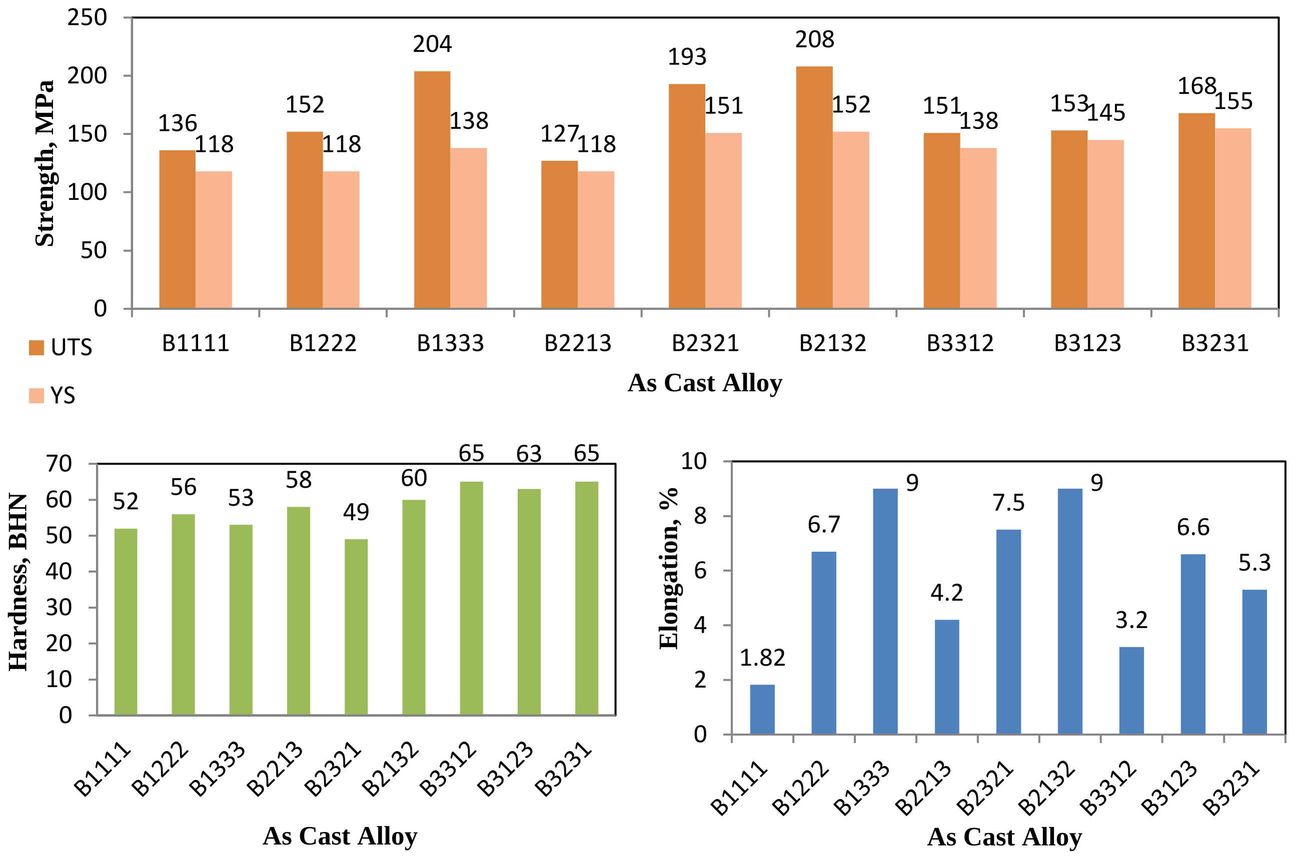


Fig. 8. Mechanical properties of the as-cast AZ80 alloys

Hence it can be concluded that B2132 and B1333 alloys which consist of thermally stable, high melting point temperature compounds in precipitate form inside the grains and along the grain boundaries can be used in light weight and moderate temperature applications that require 130 to 200 MPa strength at room temperature.

4. CONCLUSIONS

To study the combined influence of Sn and trace elements of REs and Ca on the characteristics of as-cast AZ80 alloy, an experimental investigation using nine alloys is carried out through casting route. The important observations made are:

1. With the addition of Sn, the room temperature tensile strength of AZ80 alloy is enhanced due to the presence of fine precipitates of Mg_2Sn along the grain boundaries and within the grains except for B2213 alloy. For B2213 alloy the reduction of tensile strength is observed which is attributed to its coarse grain structure and high-volume fraction of the Mg-Sn-Ca phase. Furthermore, for the alloy containing 6 wt.% of Sn, 0.9 wt.% of RE and 0.5 wt.% of Ca, better enhancement of 53 % ultimate tensile strength is noted when compared to AZ80 alloy.
2. Without Sn addition, better properties are observed for the alloy containing 1.5 wt.% RE and 0.9 wt.% Ca i.e., an improvement of 50 % in tensile strength and 394 % in elongation is observed when compared to AZ80 alloy. This is due to the formation of thermally stable, high melting point temperature Al_2(RE, Ca) precipitates.
3. Hence the co-addition of Sn, Ce, Gd &Ca in AZ80 alloy lead to refinement of microstructure in all as-cast alloys and changed the mechanical properties of AZ80 magnesium alloy significantly.

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