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RESEARCH ARTICLE

INFLUENCE OF Ti MICROALLOYING ON ZAMAK-5

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ABSTRACT

ZAMAK-5 is a Zn-Al alloy which contains 3.9-4.3 % Al, 0.75-1.25 % Cu and 0.03-0.06 % Mg. Low melting temperature and good castability are some of the advantages of ZAMAK-5. In the present study, the composition of ZAMAK-5 alloy was modified by microalloying it with Ti. Alloying was accomplished by melting ZAMAK-5 at 450 and 650 ^{o}C and introducing Ti as Al10Ti master alloy. It was found by ICP analyses that modified alloys contained 0.01 and 0.03 wt.% Ti. Re-melting and casting of the alloyed samples were conducted at 650 ^{o}C under argon atmosphere. The modified alloy that contained 0.03 % Ti had near eutectic Zn-Al composition due to the increased amount of Al. The increase in the Al content was caused by master alloy addition. According to microstructural and solidification analyses, the modified alloy that contained 0.03 % Ti had lower liquidus temperature and less primary (η) dendrites in its microstructure. Alloying with Ti was found to increase hardness and bending strength of the base alloy. Alloy that was modified with 0.03 % Ti exhibited the highest hardness (102.3±2.8 HB_{10}), compressive yield strength (290.0±5.0 MPa) and bending strength (661.4±30.5 MPa).

Keywords: *ZAMAK-5, Ti addition, Zinc-aluminum alloys, Casting.*

1. INTRODUCTION

Zn-Al alloys have been used in automotive applications, mechanical parts, and decorative areas due to their advantages such as good castability, long mold life and low melting point [1, 2]. They exhibit superior properties at cutting, drilling, reaming, tapping, turning, etc. and provide ease of use at machining applications due to advantages such as surface quality after heat treatment, low cutting forces and wear [3]. Nowadays, ZAMAK-2, ZAMAK-3 and ZAMAK-5 are the most popular Zn-Al alloys. ZAMAK-5 is a Zn-Al alloy which contains 3.9-4.3 % Al, 0.75-1.25 % Cu and 0.03-0.06 % Mg. ZAMAK-2, ZAMAK-3 ve ZAMAK-5 alloys have hypo-eutectic composition according to the Al-Zn phase diagram [4].

Azizi et. al. (2015), mechanically alloyed ZAMAK-2 via powder metallurgy (PM) method and a maximum hardness value of 101 HB was obtained. In the study, die cast and sand-cast product hardness values were compared to the hardness values of the PM products. It was found that while die cast samples exhibited better hardness value (130 HB) than the PM products, hardness values of the sand-cast samples (85 HB) were lower than the PM products [5].

Altınsoy and Kızılarslan (2016), investigated the effect of the Ti addition to ZAMAK-3 on microstructure, hardness and fracture toughness. They suggested that the amount of eutectic structure increased when Ti amount increased in the composition. Furthermore, it was reported that Ti addition did not affect the hardness and fracture toughness values of the main alloy (ZAMAK-3) [6].

Sandlöbes et. al. (2016) conducted studies to understand the aging processes of the (Zn4.3Al0.59Cu0.31Mg) Zn-Al-Cu-Mg alloys which contain almost the same amount of Al, Cu and Mg in their compositions as ZAMAK-5. In the study, tensile strength of the alloys was investigated at room temperature and at 85 ^{o}C. It was reported that the alloys exhibited better tensile strength (157 MPa) at 85 ^{o}C, than that at room temperature (133 MPa) [7].

Wu et. al. (2016), determined that increasing Mg amount in the Zn-Al alloys resulted in precipitation of the Mg_2Zn_{11} phase in a lamellar fashion in the eutectoid structure. Moreover, it was indicated that, Zn-Al alloys which had moderate amount of Mg (0.21 wt.%), had the highest yield strength at a room temperature and at high temperatures [8].

Recently, there have been studies on modifying Zn-Al alloys with different elements such as B, Ni, Mn, by different researchers [9-11]. Ayday et al. [9] examined the effects of boron addition on mechanical and thermophysical properties of ZA12 alloy. It was reported that B addition enhanced hardness and tensile strength, along with ductility. Moreover, thermal conductivity of ZA12 alloy was seen to decrease by B addition [9]. Ti was added previously to Zn-Al alloys to modify the structure [12]. Ti has limited diffusion rate in Zn and it has anti-grain growth effect on as cast Zn. [13]. It was reported that, Ti addition improved strength of pure Zn-Al alloys. In addition, it increased creep resistance of Zn alloys [14].

Although there are remarkable studies in the literature that investigate the microalloy additions to ZAMAK and Zn-Al alloys, no study that investigated the effect of the Ti addition to the ZAMAK-5 alloy was encountered. Ti has a limited solubility in Zn and forms Zn-$TiZn_{15}$ eutectic at %0.11 Ti in Zn. It is expected that $TiZn_{15}$ intermetallic plays a role in increasing the hardness and strength of the zinc alloys [4,14]. In the studies of of Türk (1996) [15] and Durman (1996) [16], 0.01 % Ti addition was reported to increase the strength and 0.03 % Ti addition was reported to increase the hardness of ZA-8 alloy. Therefore, in the present study, chemical composition of commercial ZAMAK-5 was modified by microalloying it with Ti in the vicinity of these percentages. Solidification behavior, microstructure, hardness, bending and compressive strength of the modified alloy were investigated.

2. MATERIAL AND METHODS

2.1. Preperation of Alloys and Casting

Commercial ZAMAK-5 (3.5-4.5 %Al, 0.75-1.25 % Cu, 0.03-0.08 % Mg) was alloyed with Al10Ti master alloy at 450 and 650^{o}C. The percentages given in this text are weight % (wt.%). It can be seen in the Zn-Al phase diagram that there is a eutectic melting at 380 ^{o}C at 5 wt.% Al composition [4]. In the vicinity of this composition, the alloys are completely liquid state above 400 ^{o}C. For this reason, for microalloying of ZAMAK-5 the first selected temperature was 450 ^{o}C. It was reported in literature that the alloying elements deteriorate when ZAMAK alloys are heated to above 650 ^{o}C [17].

Therefore, for microalloying of ZAMAK-5 the second selected temperature was 650 ^oC, which can be suggested as the highest allowable temperature.

Two sets of Ti microalloyed ZAMAK-5 samples were prepared. In the first set, calculated amount of Al10Ti to yield 0.05 % Ti was mixed with the ZAMAK-5 melt at 450 ^oC. In the second set, calculated amount of Al10Ti to yield 0.10 % Ti was mixed with the ZAMAK-5 melt at 650 ^oC. Alloying process was conducted in a Protherm muffle furnace for 6 hours. Obtained Ti modified alloys were cast into a steel mold having 14 mm diameter.

For the preparation of the samples, which were utilized in the microstructural examinations and in the mechanical tests, the alloys were re-melted at 650 ^oC and cast in an induction furnace (Indutherm MC 20 V, max. Power 3.5 kW). Re-melting/casting was applied for reducing porosity and obtaining better surface quality of the test samples. The utilized induction furnace contained a tiltable melting/casting chamber with protective atmosphere. There were the induction coils in which the melting crucible was placed inside the chamber. A steel mold (8 mm inside diameter) which was preheated to 150 ^oC was also placed inside the chamber, in line with the crucible. Two consecutive vacuum-argon filling cycles were applied prior to melting the alloy. Melting was conducted in argon atmosphere at 1 atm. Casting was accomplished by tilting the chamber 90 degrees so that the molten metal was poured from the melting crucible into the mold inside the chamber. The furnace was programmed so that the argon pressure inside the chamber increased to 3 atm. when the chamber was tilted for casting. In order to avoid adhesion between mold and the molten alloy, inner side of the mold was coated with hexagonal boron nitride spray (Ekamold). Same casting process was applied to non-modified base alloy (ZAMAK-5) for comparison of the results.

2.2. Microstructural and Elemental Analyses

Cast samples were cut and ground with 800, 1200, 3000 grit emery paper, polished with 1-micron diamond paste and etched with nital (2 % nitric acid in ethanol) solution. Microstructure was investigated by optical and scanning electron microscopy (SEM). An optical microscope (Nikon Eclipse LV150) and Clemex image analysis software was used for microstructural analyses. The secondary dendrite arm spacing (SDAS) size values were obtained by the measure tool of the software. The SDAS sizes were determined according to method E, which was described in [18]. In this method, SDAS sizes were determined by measuring the distance from center to center of two neighboring secondary dendrite arms. Average and standard deviation of 100 measurements were reported. In addition, the areal fractions of the phases or structures were measured by the area measurement tool of Clemex software. FESEM-EDS analyses were done by ZEISS- SUPRA 40VP.

For elemental analysis, 2 g of sawdust was removed from the samples and ICP-OES (Inductively Coupled Plasma - Optical Emission Spectrometry) analyses were performed at Meta Nickel Cobalt Company, Manisa Gördes Plant.

2.3. Mechanical Testing

Three-point bending and compressive strength tests were carried out by Shimadzu Autograph AG-IC model 50 kN universal mechanical tester. The diameters of the samples which were utilized for three-point bending tests were 8 mm. In bending tests, a span length of 25 mm was applied. Three-point bending tests were conducted according to the procedure in ASTM B528-05 standard. The diameter and the height values of the samples which were used for the compression tests, were 8 mm and 15mm, respectively. To calculate the compressive yield strength of the samples, 0.2 % offset method

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was used according to E9- 89a ASTM standard. It is indicated in this standard that the compressive strength of the ductile materials can be determined from the stress-strain diagram at a specified total strain. Therefore, in the present study, compressive strength values of the samples were determined at 50% strain and they were reported as compressive strength at 50% strain. 50% strain was selected since after this value the stress-strain curve was seen to lose linearity. Hardness tests were performed by Brinell hardness tester (Bulut Makina – Digirock) with 2.5 mm diameter steel ball tip and 62.5 kg load (HB10 scale).

2.4. Cooling Curves
In order to investigate effect of Ti addition on solidification, a set of samples was cast into a sand mold (Heraeus - Quick Cup) having 35mm x 35mm bottom sizes and 45mm height. During cooling and solidification of the samples, temperature was measured with a K-type thermocouple that was placed in the middle of the sand mold. Cooling curves were obtained by recording the temperature data via an ORDEL PC991 step control device and ORDEL SBA200 converter interface.

3.RESULTS AND DISCUSSION

3.1. Microstructural and Elemental Analyses
According to the ICP-OES analyses, 0.05 % Ti added alloy (alloyed at 450 ^{o}C) had 0.01 % Ti, and 0.10 % Ti added alloy (alloyed at 650 ^{o}C) contained 0.03 wt. % Ti in its composition (Table 1). It was determined that modified alloys contained lower quantity of Ti than the added amount. This can be originated due to the retaining of some of the added alloy in the slag. Amount of Ti in the modified ZAMAK-5 was seen to increase with the increase in the master alloy amount and alloying temperature. In accordance with the ICP-OES analysis results, ZAMAK-5 alloy that contained 0.01 wt. % Ti was coded as Z5-0.01Ti and the alloy that contained 0.03 wt. % Ti was coded as Z5-0.03Ti.

The amount of Al in the modified ZAMAK-5 was seen to increase with the increase in the master alloy amount. It was determined that Z5-0.01Ti contained 4.43 wt. % Al and Z5-0.03Ti contained 5.1 wt. % Al (Table 1). The Al amounts in the modified ZAMAK-5 samples were near the eutectic composition (5.0 %).

Table 1. Amounts of Ti and Al in the samples, according to ICP-OES analyses.

Alloy/ Element (Wt.%)	**ZAMAK-5**	**Z5-0.01Ti**	**Z5-0.03Ti**
Al	4.43	4.57	5.10
Ti	-	0.01	0.03

The eutectic Zn-Al alloys solidifies at 382 ^{o}C by eutectic transformation (L $\rightarrow$ η + β). On the other hand, in the hypo-eutectic base alloy (ZAMAK-5), primary η dendrites are formed in the liquid first. Later on, L $\rightarrow$ η + β eutectic transformations takes place. Lamellar eutectic phase (η + β) is formed around the η dendrites via eutectic transformation. After that, β in the eutectic structure transforms to Zn rich (η) phase and Al rich (α) phases via eutectoid transformation that occurs at 275 ^{o}C (transformed eutectic). As a result of the eutectoid transformation there are primer η dendrites and (η + α) phases (transformed β) in the microstructure at a room temperature. According to the EDS analyses and results in the literature, the light colored areas are primary η dendrites (shown as A in

Figure 1(b)) and dark colored areas are transformed eutectic η + α phases (shown as B in Figure 1(c)) in the microstructure of the samples given in Figure 1 [4,8]. A greater amount of η + α phase mixture was detected visually in the microstructure of the Z5-0.03Ti. This is believed to be a result of its composition being in close to the eutectic composition (Figure 1(c)). The fraction of primary η dendrites in the microstructure of the base ZAMAK-5 and Z5-0.01Ti was higher than that in Z5-0.03Ti, because of their hypo-eutectic compositions (Figure 1 (a, b)). According to the areal calculations performed through the image analyses of the microstructures given in Table 2, the area covered by the pre-eutectic η phase was 67% in base ZAMAK-5, whereas it decreased to 38 % in Z5-0.03Ti sample (the amounts of transformed eutectic structure were complementary of these values to 100%).

Effect of Ti addition on secondary dendrite arm spacing (SDAS) values of η dendrites were measured during the image analyses of the microstructures by Clemex software. The results are presented in Table 2. The SDAS values in ZAMAK-5 and Z50.01Ti sample were similar, whereas there was a decrease in the SDAS values of Z50.03Ti sample. Addition of Ti appears to have resulted in a decrease in the SDAS values of ZAMAK-5. After alloying, all the samples which were subjected to microstructural examinations and mechanical tests were obtained by melting at 650 ^{o}C and then casting. Therefore, the same cooling rate was accomplished in all the samples. As a result, the cooling rate did not have an effect on the differences in the microstructure of the samples. All the differences in the microstructure can be attributed to the effect of alloying. The reduction in the SDAS size as a result of the addition of Al10Ti master alloy may be related with the increase in the Al content of the alloy or the presence of Ti (Table 1).

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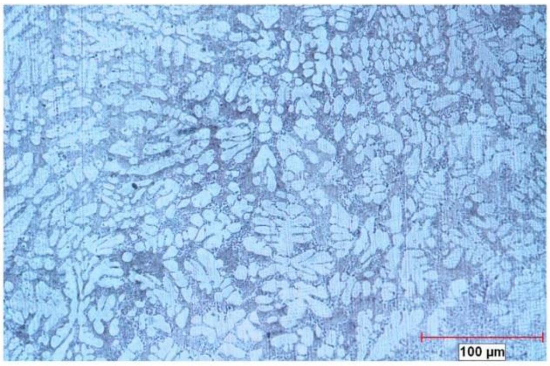
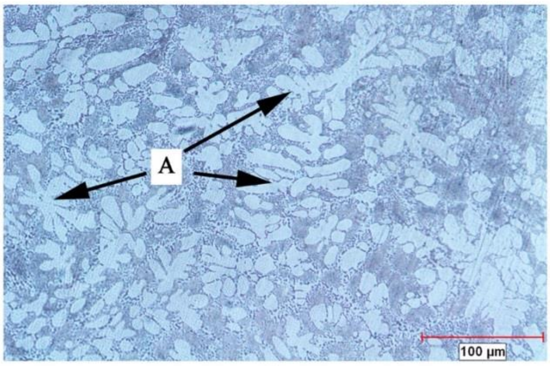
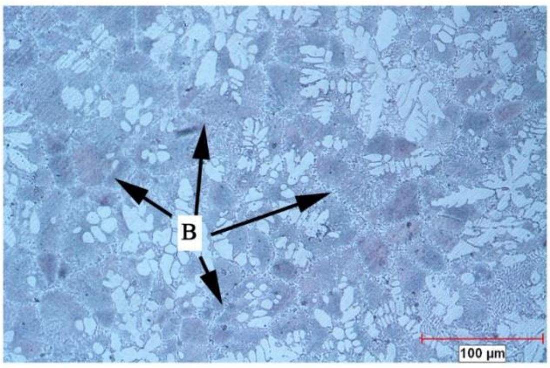


(a)



(b)



(c)

Figure 1. Microstructure images of the samples (Optical Microscope, magn. 200X), (a) Base alloy, (b) Z5-0.01Ti (Alloyed at 450 ^{o}C and re-melted and cast at 650 ^{o}C), (c) Z5-0.03Ti (Alloyed at 650 ^{o}C and re-melted and cast at 650 ^{o}C).

Table 2. Secondary dendrite arm spacing (SDAS) and area percentages of the structure in the microstructure of the samples.

Sample	SDAS (µm)	Area %	
		Pre-eutectic η	(Transformed) Eutectic Structure
ZAMAK-5	7.8±1.16	67	33
Z5-0.01Ti	7.6±1.20	56	44
Z5-0.03Ti	6.5±1.13	38	62

The magnified portion of a transformed eutectic region (which was marked as B in Figure 1(c)) is presented in Figure 2. According to the SEM-EDS analyses that was conducted on this region (Figure 2), 5 times more Ti than the overall composition was detected (Table 3). Therefore, it is considered that Ti was mostly located on the eutectic regions. This finding is in agreement with the data given in the literature, since Ti has a limited solubility in Zn [4,14].

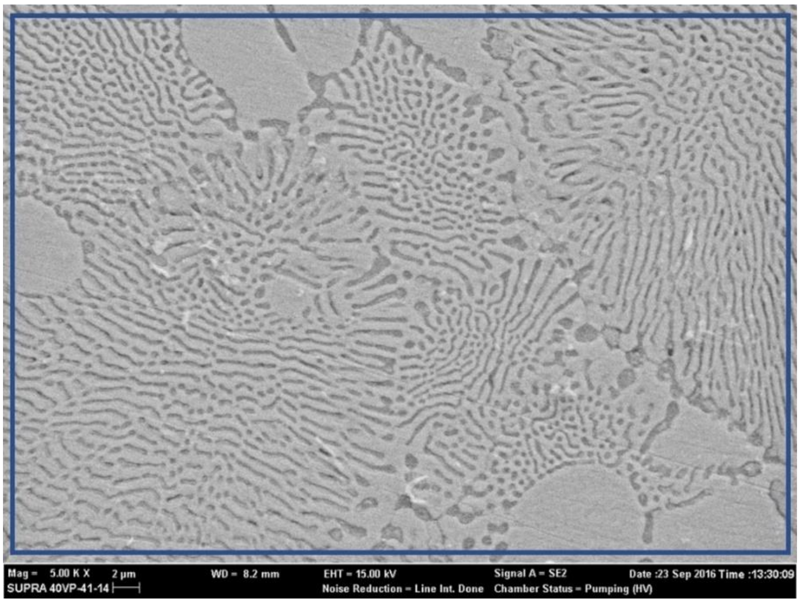


Figure 2. SEM image of transformed eutectic region of 0.03 % Ti modified ZAMAK-5 (magn. 5 kX).

Table 3. EDS result of the Ti modified ZAMAK-5 given in Figure 2 (transformed eutectic region).

	Wt. % Al	Wt. % Cu	Wt. % Ti	Wt. % Zn
Selected Region (Blue Frame)	7.49	1.25	0.15	91.11

3.2. Solidification Process of the Ti Modified and Base ZAMAK-5 Alloys

When solidification curves were examined, it was determined that the liquidus temperatures of the Ti modified alloys are lower than that of base ZAMAK-5 alloy (Figure 3). During solidification, primary η phase start to occur at 389 ^{o}C for ZAMAK-5, at 386 ^{o}C for Z5-0.01Ti and at 384 ^{o}C for Z5-0.03Ti samples. These values are in accordance with the Al contents of the samples.

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It can be seen at Figure 3. that, Z5-0.03Ti has the lowest liquidus point. This validates the less amount of the primer η dendrites formed in this sample. This result is in accordance with the microstructure images (Figure 1). In addition, it might be suggested that Z5-0.03Ti has the shortest transformation time, which is caused by its near eutectic composition.

It was determined that the solidus temperatures of all the samples were at 380-381 ^{o}C. These values agree well with the data given in the literature, since the eutectic temperature of Zn-Al alloys is at 380 ^{o}C [4].

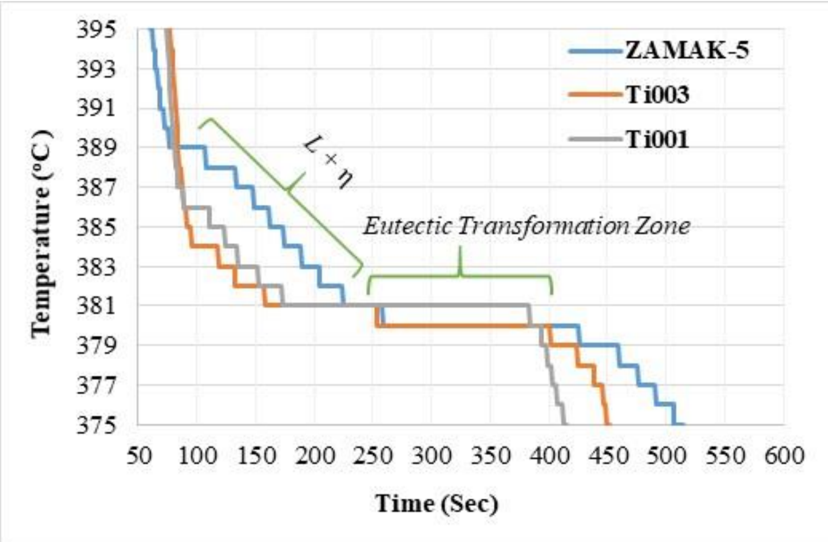

Figure 3. Cooling curves of the samples.

3.3. Hardness Test Results

According to the hardness test results, it was determined that hardness values of Ti modified alloys were higher than that of base ZAMAK-5 alloy (Figure 4.). It can be suggested that Ti presence in the eutectic regions in the microstructure may be effective on these results. In addition, the higher fraction of the eutectic structure in the of the Ti modified samples may be suggested for enhancing the hardness. While hardness value of the base ZAMAK-5 alloy was 96.9±2.4 HB_{10}, hardness of Z5-0.01Ti was 100.2±1.8 HB_{10} and that of Z5-0.03Ti was 102.3±2.8 HB_{10}.

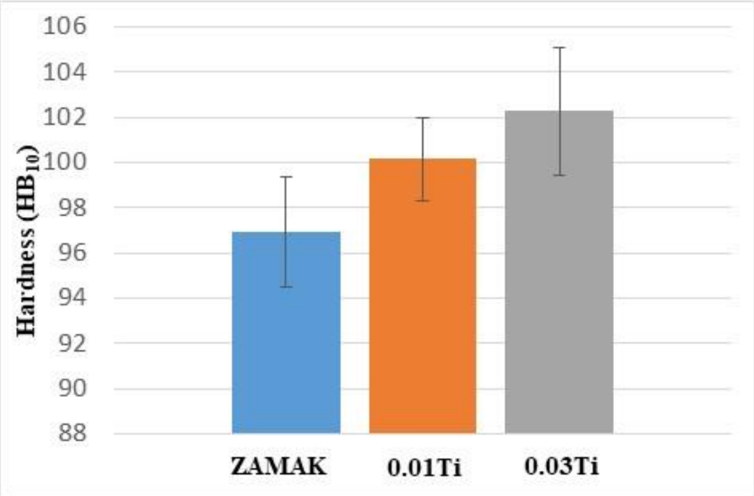

Figure 4. Hardness values of the samples.

3.4. Three-Point Bending Strength

Average and standard deviation values of the bending test results are presented in Figure 5 and Table 4. These values were obtained by averaging the results of 3 tests. It can be seen that Ti addition results in a slight difference in the 3-point bending stress-strain curves of the samples. Bending strength was determined as the highest stress attained in the bending test. Base sample and Z5-0.01Ti exhibited similar bending strength values. Bending strength of Z5-0.03Ti was higher than the bending strength values of the other samples. In the study of Ayday, ZA12 zinc alloy was microalloyed with B. The increase in the hardness and strength was attributed to the finer structure that was formed upon microalloying with B [9]. In the present study, the increase in the bending strength can be attributed to the decrease in the SDAS values and also to the increase in the amount of the eutectic structure, as a result of Al10Ti master alloy addition, as shown in Section 3.1. In addition, formation of $TiZn_{15}$ phase may have played a role in increasing the hardness and strength, as suggested in the literature [4,14]. However, this phase was not detected in the present study, most probably due to its low amount. Strain at maximum stress values appeared to be not dependent on the Ti amount of the samples.

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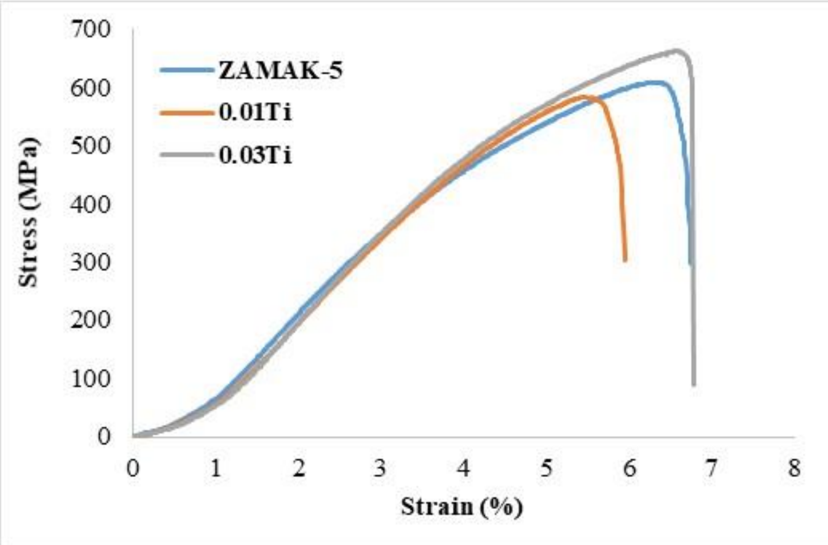

Figure 5. Stress-strain curves of the samples obtained by 3-point bending tests.

Table 4. Three-point bending strength and strain values of the samples.

Sample	Bending Strength (BS) (MPa)	Standard Deviation (BS)	Strain at Max. Stress (SaMS) (%)	Standard Deviation (SaMS)
ZAMAK-5	608.0	31.0	6.4	0.9
Z5-0.01Ti	583.3	48.4	5.5	1.3
Z5-0.03Ti	661.4	30.5	6.6	0.6

3.5. Compressive Strength

Average and standard deviation values of the compression test results are presented in Figure 6 and Table 5. These values were obtained by averaging the results of 3 tests. Compressive strength values of the samples were determined as described in E9- 89a ASTM standard. In the present study, they were determined at 50% strain from the stress-strain curves given in Figure 6 and reported as compressive strength at 50% strain in Table 5. Base alloy and Z5-0.01Ti sample exhibited similar values of 729.7±8.0 and 719.6±11.9 MPa, respectively. Z5-0.03Ti sample had a compressive strength at 50% strain value of 779.8±7.5 MPa.

Compressive yield strength values of the samples were determined according to 0.02% offset method described in E9- 89a ASTM standard by using the enlarged initial portion of the stress-strain plot given in Figure 6. Z5-0.01Ti and Z5-0.03Ti had similar compressive yield strength (about 290MPa) which was higher than that of ZAMAK-5 (about 280MPa) (Figure 6 and Table 5). It can be considered that Ti presence provides this slight increase in the compressive strength. The higher slope of the curves in the Ti modified samples can be taken as an indication of higher elastic modulus of these samples, as compared to base ZAMAK-5.

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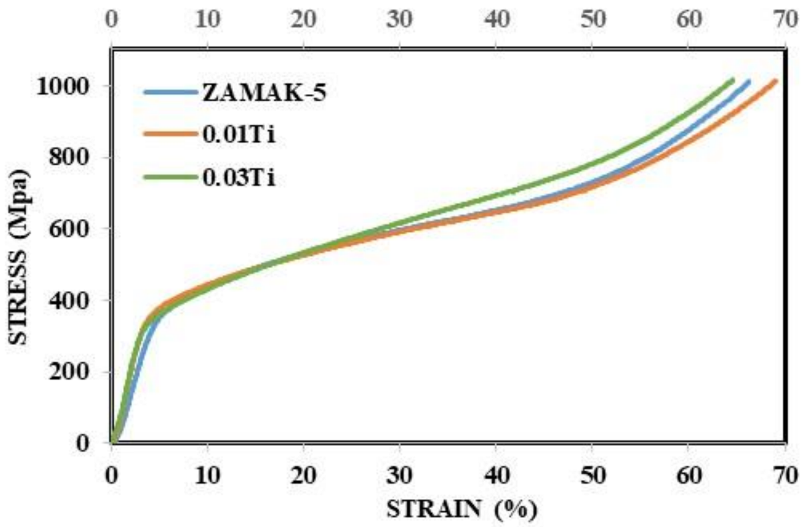

Figure 6. Stress-strain curves of the samples obtained by compression tests.

Table 5. Compressive yield strength and compressive strength at 50% strain values of the samples.

Sample	Compressive Yield Strength (CSY) (MPa)	Standard Deviation (CSY)	Compressive Strength at 50% Strain (CS50) (MPa)	Standard Deviation (CS50)
ZAMAK-5	280.3	4.5	729.7	8.0
Z5-0.01Ti	289.6	2.6	719.6	11.9
Z5-0.03Ti	290.0	5.0	779.8	7.5

4. CONCLUSION

In this study, effects of Ti microalloying on solidification temperature, microstructure, hardness, and bending and compressive strength of commercial ZAMAK-5 alloy were investigated. Modified alloys contained lower quantity of Ti than the added amount. Ti in the modified ZAMAK-5 was seen to increase with the increase in the added master alloy amount and alloying temperature. Liquidus temperature was 389 ^{o}C for ZAMAK-5, 386 ^{o}C for Z5-0.01Ti and 384 ^{o}C for Z5-0.03Ti samples. The amount of eutectic structure in the microstructure of the samples was found to increase with increasing amount of Al10Ti master alloy addition. The Al content of the samples was believed to be effective on the liquidus temperatures and on the amount of eutectic structure. As a result of EDS analyses, it was considered that Ti was mostly located on the eutectic regions. Addition of Ti resulted in a decrease in the SDAS values of ZAMAK-5. Z5-0.03Ti sample had higher bending strength than the other samples. Hardness and compressive strength values of samples which contained Z5-0.03 % Ti were higher than those of the base alloy. Obtained results show that with Ti-Al modification, ZAMAK-5 may be used for applications that require higher hardness and strength.

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