



Article Microstructure Evaluation, Quantitative Phase Analysis, Strengthening Mechanism and Influence of Hybrid Reinforcements (β-SiCp, Bi and Sb) on the Collective Mechanical Properties of the AZ91 Magnesium Matrix

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Copyright: © 2021 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https:// creativecommons.org/licenses/by/ 4.0/). Abstract: Gravitational melt-stir casting produced hybrid nano-reinforcements (β-SiCp) and microreinforcements (Bi and Sb) of AZ91 composites. SiCp-diffused discontinuous β -Mg₁₇Al₁₂ precipitation with a vital factor of SiC was exhibited at the grain boundary region, formulated Mg₃Si throughout the composite and changed the present Mg_{0.97}Zn_{0.03} phases. The creation of Mg₂Si (cubic) and SiC (rhombohedral axes) enhanced the microhardness by 18.60% in a 0.5 wt.% SiCp/AZ91 matrix. The microhardness of 1 wt.% SiCp/AZ91 was slightly reduced after $Mg_{0.97}Zn_{0.03}$ (hexagonal) reduction. The best ultimate tensile value obtained was about 169.33 MPa (increased by 40.10%) in a 0.5 wt.% SiCp/AZ91 matrix. Microelements Bi and Sb developed Mg3Bi2, Mg3Sb2 and monoclinic C_{60} phases. The best peak yield strength of 82.75 MPa (increased by 19.85%) was obtained with the addition of 0.5 wt.% SiCp/1 wt.% Bi/0.4 wt.% Sb. The mismatch of the coefficient of thermal expansion of segregated particles and the AZ91 matrix, the shear transfer effect and the Orowan effect, combined with the quantitative value of phase existence, improved the compressive strengths of the composites with 0.5 wt.% β -SiCp, 1 wt.% β -SiCp and 0.5 wt.% SiCp/1 wt.% Bi/0.4 wt.% Sb by 2.68%, 6.23% and 8.38%, respectively. Notably, the Charpy impact strengths of 0.5 wt.% and 1 wt.% β-SiCp-added AZ91 composites were enhanced by 236% (2.89 J) and 192% (2.35 J), respectively. The addition of Bi and Sb with SiCp resulted in the formation of a massive phase of brittle Al_6Mn . Al-Mn-based phases (developed huge voids and cavities) remarkably reduced impact values by 80% (0.98 J). The discussion covers the quantitative analyses of X-ray diffraction, optical microscopy and scanning electron microscopy results and fracture surfaces.

Keywords: magnesium alloy; grain and interface; composite; mechanical behaviour; Orowan effect; strengthening mechanism; intermetallic phase; quantitative phase analysis; fractography

1. Introduction

The engineering sector (commercial and military aerospace vehicles, automotive industry and shipbuilding industry) has recently shown broad interest in magnesium alloys [1–4] because of their light weight, which reduces fuel consumption and green gas emission [5]. Magnesium alloys and their composites have low weight (density $\rho = 1.74$ g/cm³), high specific strength (158 KN m/kg) and superior mechanical strength [6]. Nevertheless, the applications, quality and quantity of magnesium alloys are limited [7] because of the constrained slip arrangement of the hexagonal closed-packed (HCP) crystal lattice [8]; thus, their tensile, compressive and impact properties must be improved. The Federal Aviation Association (FAA) lately reconsidered the practicability of using WE43 and Elektron 21 magnesium alloys because of the increasing demand for novel magnesium alloys. The FAA removed the prohibition against the use of magnesium alloy in seat frame compartments [9].

Thus, magnesium alloys with different nano- and microparticles need to be investigated through different mechanical tests.

Several studies have investigated magnesium metal matrix composites (MMCs) to enhance their mechanical properties, such as tensile [10], compression [11] and impact properties [12]. Many factors influence the strength of materials, such as the method of fabrication (ultrasonic stirring method) [13], extrusion of composite and extrusion speed [14], variation in extrusion temperature [13], ageing behaviour [15], variation in ageing temperature [16], annealing behaviour of composites [6] and size of reinforcements [17,18] (microelements and nanoparticles). However, the tensile, compression and impact properties of the hybrid β -SiCp nanoparticle and Bi/Sb microelement reinforcements of AZ91 have not been studied by many. Moreover, the scattering of reinforcements is vital to amend a composite's mechanical strength at a low cost [19,20].

The present paper investigated the hybrid reinforcements of AZ91 (β -SiCp, Bi and Sb) in the as-cast condition by the gravitational melt-stir casting method. The aim of adding SiCp was to settle in the grain boundary and suppress the discontinuous β -Mg₁₇Al₁₂ precipitation of the AZ91 matrix [21]. In addition, the addition of Bi and Sb formed Mg₃Bi₂ and Mg₃Sb₂ phases, which acted as a straddle in the AZ91 matrix and improved the composite's mechanical properties [22,23]. The mechanical properties of the AZ91 composites with β -SiCp, Bi and Sb were studied by tensile, compression and Charpy impact tests.

The microstructure and phase changes in the composites were studied by X-ray diffraction (XRD), scanning electron microscopy (SEM) and electron-dispersive X-ray spectroscopy (EDS). The qualitative and quantitative phases in the matrix and strengthening mechanisms, such as the CTE, the shear transfer effect, the Orowan effect and fracture surfaces, of all tested samples were studied in detail.

2. Experimental

2.1. Experiment Materials

The present study was conducted on AZ91 magnesium matrix composites reinforced with β -SiCp with different weight percentages (50 nm average particle size), bismuth (50 μ m) and antimony (150 μ m). The nominal chemical composition of the mother alloy that conforms to AZ91 is shown in Table 1. The same patch production material was used in this study [17,24]. Guangyu Technology Co., Ltd., (Shenzhen, China) and Jiehan Technology Corporation (Taichung) from Taiwan supplied commercial AZ91 alloy and β -SiCp nanoparticles (>99.99% high purity), respectively. Johnson & Annie Co., Ltd., New Taipei, Taiwan, provided bismuth and antimony (>99.99% high-purity) reinforcements.

 Table 1. Nominal chemical composition of the investigated AZ91 alloy.

Elements	Al	Zn	Mn	Fe	Si	Cu	Ni	Mg
Wt.% Concentration	8.95	0.84	0.26	0.005	0.009	0.0008	0.0007	Balance

2.2. Composite Preparation

The different composites shown in Table 2 were produced by gravitational melt-stir casting with suitable flux-covered protective atmospheres in a mild steel crucible, as shown in Figure 1.

Table 2. Weight percentages of the investigated composites with reinforcements and their codes.

Commonito		Wt.% of Reinforcements			
Code	AZ91	β-SiCp (50 nm)	Bismuth (Bi) (50 μm)	Antimony (Sb) (150 µm)	
Code 1	100	0	0	0	
Code 2	99.5	0.5	0	0	
Code 3	99	1	0	0	
Code 4	98.1	0.5	1	0.4	



Figure 1. Gravitational melt-stir casting setup and main apparatuses.

Initially, the melts were rapidly heated at 760 °C and the temperature was maintained for 20 min. Then, these melts were stirred for 10 min at 1000 r/min to ensure uniformly dispersed reinforcements in the magnesium composites [12]. After each composite was fully melted, the melts were poured into closed moulds. During the process, the chamber was set up with mixed atmosphere gases of SF₆, CO₂ (1 vol.% and balance) and Ar at 400 °C and 700 °C to terminate oxidation and burning [22].

2.3. Microstructure

According to the American Society for Testing and Materials (ASTM) E3-11 standard of metallographic procedures [25], cubic specimens (10 mm \times 10 mm \times 10 mm) were sectioned and cut from the centre of the castings using an abrasive cut-off machine. Subsequently, these samples were mechanically ground with silicon carbide polishing papers of various grit sizes (400, 1000, 2000 and 4000 CW) and polished with diamond paste paper (0.3 µm) using water as a lubricant. Before the microstructural investigation, the polished samples were etched for 50 s with an etching solution (75 mL of ethanol, 25 mL of deionized H₂O, 1 mL of nitric and acetic acids). The microstructure and microscopic morphology were analysed by optical microscopy (OM), SEM (JSM-6390L, JEOL, Tokyo, Japan) and EDS. Phase investigation was carried out for all alloy codes by EDS and XRD (Bruker D2 phaser, Billerica, MA, USA) with CuK α radiation at 45 Kv and a current strength of 0.8 mA for composition determination. Measurements were conducted from 20° to 80° at a scan rate of 5°/min with a 12 min scanning span.

2.4. Mechanical Test at Room Temperature

2.4.1. Harness Test

Vickers microhardness was measured in different regions of each specimen using an Akashi MVK-H1 Vickers hardness tester (Mitutoyo, Kawasaki, Japan) with diamond indentation (2.5 mm diameter, 300 gmf applying a load, 10 s dwell time). The microhardness value was calculated based on the average of 10 indentations with two samples.

2.4.2. Tensile Test

The dimensions and location of the dog bone cross-section of the tensile nanocomposite sample are shown in Figure 2. The samples were cut by a water jet cutting machine as per the ASTM E8 standard [26]. Tensile tests were performed on an 810 kN MTS Insight universal testing machine at a constant speed of 0.5 mm/min at room temperature. The alloy composite's mechanical properties (yield strength, ultimate tensile strength (UTS) and elongation) were evaluated by the average of at least three samples to ensure the precision of the final results.



(a) Tensile, compression and impact test samples location & direction.



Figure 2. Schematic illumination of the dimensions of the tensile, compression and impact samples and the direction of the alloy composites.

2.4.3. Compression Test

Three to five cylindrical compression samples (constant length-to-diameter ratio of 1:1) were obtained from the same composites at different locations, as shown in Figure 2, to evaluate the compressive mechanical properties of each composite code as per ASTM E9-19 [27]. The universal testing machine (MTS, 810 kN) (MTS, Eden Prairie, MN, USA) was used at a strain rate of 0.5 mm/min for the compressive tests to evaluate the composites' compressive behaviour at room temperature.

Flow stress (σ) and true strain (ε) values were estimated [17,28] based on the given input load (F), the radius of the cylindrical specimen (R) and the original height of the sample (l_0) in the testing machine according to Equations (1) and (2):

$$\sigma = \frac{F}{\pi R^2} \tag{1}$$

$$\varepsilon = \frac{l_0 - l}{l_0} \tag{2}$$

2.4.4. Impact Test

Charpy impact test specimens (55 mm \times 10 mm \times 10 mm) were cut and evicted from different regions in each composite by a water jet cutting machine. Moreover, a CNC CHMER electrical discharge machining (CW-43CS, CHMER, Taichung, Taiwan) tool was used to make a V notch, as shown in Figure 2. Three samples were measured to evaluate the precise impact values. The samples were studied with a universal impact tester (model 74) as per ASTM E-23 [29]. All the testing specimens were polished well before testing to terminate uneven alignment, which could lead to improper results. SEM was employed to analyse all fracture surfaces after completing the impact test.

3. Results

3.1. Microhardness

Vickers microhardness values of the hybrid nano- and micro-reinforced Mg MMCs are given in Figure 3 and Table 3. The microhardness of alloy codes 2 and 3 increased by 18.60% and 14.66% compared with the as-cast alloy AZ91, respectively, but decreased with increasing β -SiCp percentage from 0.5 to 1 wt.%. The 3' microhardness of the AZ91 alloy added with 1 wt.% β -SiCp was slightly reduced; thus, the addition of β -SiCp limited the microhardness of AZ91. The microhardness of alloy code 4 (AZ91 + 0.5 wt.% SiCp + 1 wt.% Bi + 0.4 wt.% Sb) increased by 17.49% compared with that of the pure as-cast AZ91 alloy, as shown in Figure 3 and Table 3. However, the microhardness decreased slightly for alloy code 4 (AZ91 + 0.5 wt.% SiCp + 1 wt.% Bi + 0.4 wt.% Sb) compared with alloy code 2.



Figure 3. Microhardness of AZ91 alloys with different hybrid nano- and micro-reinforcements.

Table 3. Microhardness variation and increment in microhardness of the alloy codes compared with pure AZ91 all	lloy
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Alloy Code	Composition	Microhardness (HV)	Increment in Microhardness (%)
Code 1	Pure AZ91	68.21	-
Code 2	AZ91 + 0.5 wt.% SiCp	80.90	18.60
Code 3	AZ91 + 1 wt.% SiCp	78.21	14.66
Code 4	AZ91 + 0.5 wt.% SiCp + 1 wt.% Bi + 0.4 wt.% Sb	80.14	17.49

3.2. Mechanical Properties

3.2.1. Tensile Properties

The room-temperature tensile curves and mechanical tensile properties of pure and hybrid nano-micro-reinforced (β -SiCp, Bi and Sb) as-cast composites are depicted in Figure 4. The comparison of the present study's yield strength, UTS, Young's modulus and elongation with those of other particles reinforced by MMCs in other studies is shown in Table 4. The engineering tensile properties of nanocomposites in as-cast conditions improved remarkably compared with those of the pure AZ91 counterpart.



Figure 4. Engineering stress–strain curve for AZ91 alloy composites with different hybrid nano- and micro-reinforcements.

Table 4. Tensile properties of pure AZ91 and AZ91 composites with different hybrid nano- and micro-reinforcements.

Alloy Code	Types of Castings	0.2% Yield Strength (MPa)	UTS (MPa)	Elongation (%)
Code 1	Pure AZ91	69.05 ± 2	120.87 ± 4	6.0 ± 0.1
Code 2	AZ91 + 0.5 wt.% SiCp	72.82 ± 3	169.33 ± 2	21.6 ± 0.3
Code 3	AZ91 + 1 wt.% SiCp	59.38 ± 3	118.07 ± 3	10.6 ± 0.2
Code 4	AZ91 + 0.5 wt.% SiCp + 1 wt.% Bi + 0.4 wt.% Sb	82.75 ± 1	159.60 ± 4	11.2 ± 0.2
Other study [17]	AZ91(as-cast) + 1 wt.% WS ₂	67.93 ± 3	140.81 ± 6	9.01 ± 0.5
Other study [30]	AZ91(as-cast) +1 wt.% SiCp	116	139	0.78

The variations in the mechanical properties of pure and reinforced AZ91 composites are depicted in Table 4. Tensile strength (169.33 MPa, increased by 40.10%), yield strength (72.82 MPa, increased by 5.47%) and elongation (21.6%, increased by 3.6 times) dramatically increased with an increase in β -SiCp. The increase in elongation reflects that 0.5 wt.% β-SiCp reinforcement increases the formability of the AZ91 matrix alloy substantially. When SiCp content is 0.5 wt.%, the grain refinement effect and the constructively refined Mg17Al12 phase of granulated morphology compounds cause the increase in elongation. Tensile strength (118.07 MPa, decreased by 2.31%) and yield strength (59.38 MPa, decreased by 14.01%) decreased with the addition of 1 wt.% β -SiCp, whereas elongation was preserved (10.6%, increased by 1.77 times). Furthermore, the addition of bismuth and antimony with β -SiCp in alloy code 4 increased the peak yield strength (82.75 MPa, increased by 19.85%) compared with pure AZ91 and the other alloys. In addition, UTS and elongation in alloy code 2 (159.60 MPa, increased by 32.04% compared with pure AZ91) and alloy code 3 (11.2%, increased by 1.87 times) increased compared with pure AZ91. Moreover, the present nanocomposite clearly shows a superior tensile value (UTS, enhanced by 20.25% [17]) compared with other magnesium MMCs [17,30] (such as micro-WS₂ and nano-SiCp) or large-sized particles in the as-cast condition, as depicted in Table 4.

3.2.2. Compression Properties

Figure 5 shows the room-temperature engineering compressive stress–strain curves and properties of pure AZ91 and AZ91/ β -SiCp/Bi/Sb composites in the as-cast condition. Compressive strength and ductility developed instantaneously with the addition of β -SiCp/Bi/Sb reinforcements, as depicted in Table 5. The compressive properties of alloy codes 2, 3 and 4 improved by 2.68%, 6.23% and 8.38%, respectively, compared with pure AZ91 (345.14 MPa). The present study achieved a higher compressive strength compared with other micro-SiCp-reinforced composites in similar previous studies [31].



Figure 5. Engineering compressive stress–strain curves of pure AZ91 and AZ91 composites with different hybrid nano- and micro-reinforcements.

Table 5. Compression strength comparison of pure AZ91 and AZ91 composites with different hybrid nano- and micro-reinforcements.

Alloy Code	Types of Castings	Maximum Compressive Strength (MPa)	Compression Ratio (%)
Code 1	Pure AZ91	345.14 ± 13	16.17 ± 0.3
Code 2	AZ91 + 0.5 wt.% SiCp	354.38 ± 10	15.73 ± 0.1
Code 3	AZ91 + 1 wt.% SiCp	366.63 ± 13	15.90 ± 0.3
Code 4	AZ91 + 0.5 wt.% SiCp + 1 wt.% Bi + 0.4 wt.% Sb	374.07 ± 13	15.54 ± 0.3

3.2.3. Impact Properties

The variations in the absorbed Charpy impact energy of pure AZ91 and reinforced (β -SiCp/Bi/Sb) nanocomposites are depicted in Figure 6. Table 6 shows that the absorbed energy expressively improved by 236% (2.89 J) and 192% (2.35 J) after the addition of β -SiCp reinforcements in alloy codes 2 and 3, respectively, compared with pure AZ91 (1.23 J). However, the addition of microelements (Bi and Sb) with the hybrid nanoelement (β -SiCp) in alloy code 4 decreased the impact energy by 80% (0.98 J).



Impact strength variation of AZ91 alloy codes

Figure 6. Charpy impact test and absorbed impact energy of pure and modified AZ91 alloys.

Table 6. Variations in the Charpy impact energy of pure AZ91 and AZ91/ β -SiCp/Bi/Sb composites.

Alloy Code	Types of Casting	Absorbed Energy (J)	Increment in Absorbed Energy (%)
Code 1	Pure AZ91	1.23	-
Code 2	AZ91 + 0.5 wt.% SiCp	2.89	236
Code 3	AZ91 + 1 wt.% SiCp	2.35	192
Code 4	AZ91 + 0.5 wt.% SiCp + 1 wt.% Bi + 0.4 wt.% Sb	0.98	-80

3.3. X-Ray Diffraction (XRD) Texture Analysis

Figure 7 and Table 7 show the presence of the different composition phases in hybrid nanocomposites (alloy codes 1 to 4) in the as-cast condition, which are confirmed by the XRD test results. The total weight of the phases must be equal to 1 as per Equation (3). The reference intensity ratio (RIR) method was used to estimate the quantitative value of the substantial phases according to Equation (4) [12]. RIRs are the ratios of the most substantial peak of the unknown phase to the peaks of standard phases.

$$\sum_{k=1}^{n} W_k = 1 \tag{3}$$

$$W_{\alpha} = \frac{I_{i\alpha}}{\text{RIR}_{\alpha c}} \times \left(\sum_{k=1}^{n} \frac{I_{ik}}{\text{RIR}_{kc}}\right)^{1} \times 100\%,\tag{4}$$

where $I_{i\alpha}$ and I_{ik} are the strongest intensities of the *i*-th \propto and *k* unknown phases, respectively, and RIR_{αc} and RIR_{kc} are the strongest intensity ratios of the unknown \propto or *k* phase and corundum *c*.



Figure 7. XRD pattern of as-cast AZ91 and AZ91/β-SiCp/Bi/Sb composites.

Table 7. Major intermetallic	phases with quantitati	ve values for pure AZ91 and	d reinforced AZ91 composites
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Alloy Code	Composition	Major Phases	Quantitative Values (%)	Structure
		Mg17Al12	6.81	Cubic
Code 1	Pure AZ91	Mg0.97Zn0.03	91.30	Hexagonal
		Al6Mn	1.89	Orthorhombic
		Mg17Al12	5.18	Cubic
$C = 1 \cdot 2$	$\sqrt{791} + 0.5$ wet % SiC n	Mg0.97Zn0.03	90.38	Hexagonal
Code 2	AZ91 + 0.5 Wt. // SICP	SiC	2.52	Rhombo.h.axes
		Mg2Si	1.92	Cubic
		Mg17Al12	4.64	Cubic
		Mg0.97Zn0.03	86.99	Hexagonal
C = 1 = 2	$\sqrt{701}$ + 1 wet % SiC p	SiC	2.69	Rhombo.h.axes
Code 3	AZ91 + 1 wt. // SICP	Mg2Si	2.11	Cubic
		C60	1	Monoclinic
		Al6Mn	2.57	Orthorhombic
		Mg17Al12	15.53	Cubic
		Mg0.97Zn0.03	41.37	Hexagonal
		SiC	14.15	Rhombo.h.axes
C = 1 = 4	AZ91 + 0.5 wt.% SiCp +	Mg2Si	2.03	Cubic
Code 4	1 wt.% Bi + 0.4 wt.% Sb	C60	1.31	Monoclinic
		Mg3Bi2	15.83	Hexagonal
		Mg3Sb2	4.13	Hexagonal
		Al6Mn	5.64	Orthorhombic

The diffraction peaks of solid solution of α -phase (Mg) and β -phase (Mg₁₇Al₁₂) were perceived in all the AZ91 matrix composites. The diffraction peaks of SiC, Mg₂Si and Mg_{0.97}Zn_{0.03} were found in alloy code 2. Additionally, the counts of SiC and Mg₂Si were progressively enhanced with the addition of 1 wt.% β -SiCp in alloy code 3. In other words, the formation of Mg₂Si substantially dissolved the brittle phase of Mg₁₇Al₁₂ in alloy code 2 [19,21]. The Mg₂Si phase was segregated throughout the composite based on the XRD pattern [32]. Moreover, the appearance of $Mg_{0.97}Zn_{0.03}$, SiC and Mg_2Si in magnesium composites has also been observed in previous studies [12,30,33–35].

Figure 7 portrays the phases of SiC, Mg_2Si , Mg_3Bi_2 , Mg_3Sb_2 , C_{60} , $Mg_{0.97}Zn_{0.03}$ and Al_6Mn detected in alloy code 4, and these morphologies of precipitates have been confirmed by earlier studies [12,22,36–38]. A massive amount of Al–Mn-based intermetallic phases and brittle Al_6Mn phases were dispersed throughout the casting. Furthermore, the EDS study confirmed the phases identified in the XRD patterns.

3.4. Microstructural Analysis

The OM and SEM images of the microstructures of pure AZ91 and reinforced AZ91 composites in the as-cast condition are exhibited in Figures 8–11. EDS was used to examine the presence of phases in the MMCs.



Figure 8. Optical micrographs of (**a**) pure AZ91 matrix, (**b**) AZ91 + 0.5 wt.% SiCp, (**c**) AZ91 + 1 wt.% SiCp and (**d**) AZ91 + 0.5 wt.% SiCp + 1 wt.% Bi + 0.4 wt.% Sb.



Figure 9. SEM images of (**a**) pure AZ91 matrix, (**b**) AZ91 + 0.5 wt.% SiCp, (**c**) AZ91 + 1 wt.% SiCp and (**d**) AZ91 + 0.5 wt.% SiCp + 1 wt.% Bi + 0.4 wt.% Sb.



Figure 10. EDS spectra of (a) pure AZ9 matrix, (b) AZ91 + 0.5 wt.% SiCp and (c, d) AZ91 + 1 wt.% SiCp.



Figure 11. EDS spectra of (**a**) AZ91 + 0.5 wt.% SiCp + 1 wt.% Bi + 0.4 wt.% Sb and (**b**) Si particle dispersion at the boundary region of AZ91 + 0.5 wt.% SiCp.

Alloy code 1 contains an α -Mg matrix and enormous β -Mg17Al12 intermetallic phases. Based on the EDS examination, alloy code 1 contains Mg, Al and a small quantity of Zn. Discontinuous β -Mg17Al12 precipitates arise when eutectic supersaturated Al and Mg cool down and at the eutectic Al supersaturated solution state. New Mg2Si phases developed at the boundary of alloy codes 2 to 4, in addition to the massive β -Mg17Al12 phases. The XRD patterns in Figure 7 indicate the presence of α -Mg, β -Mg17Al12 and Mg2Si. The rough grain structure of alloy code 1 (Figures 8a and 9a) was refined remarkably compared with that of alloy code 2 (Figures 8b and 9b) [39]. On the other hand, the grain sizes of alloy codes 1–4 were calculated by ImageJ software (1.53j, U.S. National Institutes of Health, Bethesda, MD, USA) and the linear intercept method. The grain sizes were estimated from the three images of each alloy code. The average grain sizes of alloy codes 1–4 were recorded as 87, 61, 47 and 81 µm, respectively. The grain refinement increased in alloy codes 2 and 3 remarkably.

In addition, illustrative SEM images (alloy code 2; Figure 11b) show the relatively good scattering of SiC nanoparticles in alloy code 2. The solid solution of discontinuous $Mg_{17}Al_{12}$, which was scattered in a sizeable free region in alloy code 1, as shown in Figure 9a, was dramatically dissolved into alloy code 2, as shown in Figure 9b.

Nevertheless, the addition of 1 wt.% β -SiCp to AZ91 (alloy code 3) can produce many Si nanoparticles and has the potential to reproduce and formulate Mg₂Si (dendritic shape structure of the brittle phase), as shown in Figure 8c. Figure 10c specifies the EDS results for alloy code 3. Alloy code 3 had enormous phases of Al (48.78 wt.%) and Mn (47.26 wt.%), which formed intermediate brittle phases, such as Al₆Mn and others present in the massive Mg₁₇Al₁₂ phase in Figure 7 and Table 7. Similarly, Figure 10d shows an enormous solid phase of Mg (52.19 wt.%) and Al (47.76 wt.%). This phase is viewed as a massive discontinuous Mg₁₇Al₁₂ phase when the β -SiCp amount is increased to 1 wt.%. Moreover, XRD studies also confirmed the existence of these phases.

The OM and SEM images of alloy code 4 (AZ91 + 0.5 wt.% SiCp + 1 wt.% Bi + 0.4 wt.% Sb) are depicted in Figures 8, 9 and 11. Phases α -Mg, Mg₁₇Al₁₂, Mg₂Si, Mg₃Bi₂, Mg₃Sb₂ and C₆₀ were identified. The OM and SEM images show that the hexagonal structures of Mg₃Bi₂ and Mg₃Sb₂ are distributed throughout the matrix as rod shapes in the boundary layers in Figures 8d and 9d. The EDS results (Figure 11a) confirmed the presence of Mg (46.7 wt.%), Bi (3.5 wt.%), Sb (1.1 wt.%), Si (0.1 wt.%) and C (20.3 wt.%) elements. Previous studies have also confirmed the creation of Mg₃Bi₂ and Mg₃Sb₂ in the massive β -Mg₁₇Al₁₂ phase [22,23,32,35–37,39,40]. The XRD texture for alloy code 4 confirmed these phases, as shown in Figure 7.

4. Discussion

4.1. The Strengthening Mechanism of Microhardness

Research analysis by different research groups [41,42] has confirmed that the content of rigid reinforcements in the ductile material matrix enhances the hardness of the composite. The present research also affirmed the same by comparing the microhardness of alloy codes 1 to 4. Each alloy had a different weight percentage and various combinations of reinforcements compared with the as-cast condition of AZ91. For alloy code 2 (0.5 wt.% SiCp added), the microhardness was enhanced by 12.69 HV because of the creation of the brittle texture of Mg_3Bi_2 , Mg_2Si (1.92%, cubic) and SiC (2.52%, rhombohedral axes (rhombo.h.axes)). SEM images (Figure 9b,c) reveal that alloy codes 2 and 3 had grain refinement and particle strengthening, which improved the hardness of the composites [43]. Similarly, the microhardness of alloy code 3 (1 wt.% SiCp added) increased by 10 HV compared with alloy code 1. However, the microhardness of alloy code 3 decreased by 2.69 HV, from 90.38% to 86.99%, compared with alloy code 2 because of the formation of $Mg_{0.97}Zn_{0.03}$ (hexagonal) [12]. The values of the quantitative phases were estimated based on the XRD pattern results, as shown in Table 7. Furthermore, the microhardness of alloy code 4 (0.5 wt.% SiCp + 1 wt.% Bi + 0.4 wt.% Sb) increased by 11.93 HV, even with the additional brittle reinforcements Bi and Sb. Here, the microhardness was reduced

because of the non-uniform distribution of different reinforcements and their high porosity (Figure 12d) [17]. The presence of reinforcements can be ascribed to the restriction of localized matrix deformation during indentation. The existence of growing agglomeration enhances the porosity concerning the high surface tension and poor wetting properties among the particles and molten melts. The mechanism of the higher viscosity of molten metals and developing propensity of particles to clump increases the agglomeration [44].



Figure 12. Fractography of the tensile-ruptured surfaces of (**a**) pure AZ91 matrix, (**b**) AZ91 + 0.5 wt.% SiCp, (**c**) AZ91 + 1 wt.% SiCp and (**d**) AZ91 + 0.5 wt.% SiCp + 1 wt.% Bi + 0.4 wt.% Sb.

4.2. Strengthening Mechanism of the Mechanical Properties

As shown in Figures 4–6, the addition of nano-micro-reinforcements to the AZ91 matrix remarkably improved the tensile, compression and impact values compared with the as-cast pure AZ91. The strengthening factors included the mismatch in the CTE, the shear transfer effect of the load, Orowan strengthening development and the quantitative phase present in the composites. These effects contributed to the improvement of mechanical properties, which have also been confirmed by previous studies [17,21,30,33,45,46].

The dislocation effect and induced residual stresses developed because of the CTE mismatch between the AZ91 matrix and reinforcements. The plastic deformation arose in the SiCp vicinity to the Mg alloy because of thermal stresses. The increase in the mechanical properties caused by the CTE can be estimated by Equation (5) [21,45]:

$$\Delta\sigma_{CTE} = \sqrt{3}\beta\mu_m b \sqrt{\frac{12f\Delta\alpha\Delta T}{(1-f)b\overline{d}}},\tag{5}$$

where β is 1.25 (strengthening coefficient), μ_m is 2.64 GPa (elastic modulus of the matrix; μ_m can be calculated by $\mu_m = E_m/[2(1 + \nu)]$, where E_m and ν are the Young's modulus and

Poisson's ratio of the matrix AZ91, respectively), b = 0.32 nm (Burgers vector) [45], f is 0.0058 (the volume fraction of particles), \bar{d} is the diameter of the particles from Table 2, ΔT is 300 K (test temperature and processing temperature difference) and $\Delta \alpha$ is 25.5×10^{-6} (CTE difference between matrix and reinforcement particles) [47]. Particle size and volume fraction are the critical factors for CTE values, as shown in Equation (5). Thus, $\Delta \sigma_{\text{CTE}}$ increased when the particle size of the reinforcements decreased. The influence on strengthening was due to the $\Delta \sigma_{\text{CTE}}$ between pure AZ91 and reinforced AZ91, as shown in Table 8.

Table 8. Percentage of strengthening contribution and comparison of the yield strengths of pure AZ91 and the as-cast AZ91 + 0.5 wt.% SiCp + 1 wt.% Sb + 0.4 wt.% Sb matrix.

Strengthening Mechanism	Values (MPa)	Strengthening Contribution (%)
$\Delta \sigma_{ m Load}$	0.17	1.712
$\Delta \sigma_{\rm CTE}$	7.67	77.24
$\Delta \sigma_{ m Orowan}$	2.09	21.05
$\Delta \sigma_{ m Total}$	9.93	-
$\Delta\sigma_{\rm Yield\ strength\ (AZ91+0.5\ wt.\%\ SiCp+0.1\ wt.\%\ Bi+0.4\ wt.\%Sb)-(Pure\ AZ91)}$	13.70	72.48

Shear load transfers from hard reinforcement particles to the rigid composites during the mechanical testing when sound structural integrity exists between the two phases in the matrix. The load transfer effect is represented by Equation (6) [17,21,33]:

$$\Delta \sigma_I = 0.5 f \sigma_m,\tag{6}$$

where σ_m is the tensile yield strength and f = 0.00283 is the volume fraction of the reinforcement particle. The calculated load transfer values are mentioned in Table 8. Load transfer is a critical factor that improves yield strength [21,46]. Increasing load transfer consequently expands the volume of fractions, as shown in Table 8.

As per the Orowan mechanism, finer reinforcements (less than 100 nm) [48] with highly dispersed particles are more practical to enhance the mechanical properties of the composite [49]. The Orowan mechanism developed by the particles is shown in Figure 13 The Orowan mechanism is large and energetically not favourable for the dislocation to cut through in the composites. However, the Orowan mechanism is not vital in micro-sized reinforcements in magnesium MMCs because of the coarse texture and the large spacing of interparticles [48]. As shown in Table 8, the Orowan mechanism contributed about 21.05% against the CTE effect. The collaboration of the non-clipping particles remarkably increased the composite's strength as per the effect of the Orowan strengthening mechanism. The $\Delta \sigma_{\text{Orowan}}$ caused by the second-phase dispersion [50] that acts as uniformly distributed spherical reinforcements in the matrix can be estimated by Equations (7) and (8) [30,46,47]:

$$\Delta \sigma_{Orowan} = \frac{0.13G_m b}{\lambda} \ln \frac{d_p}{2b},\tag{7}$$

$$\lambda = d_p \left[\left(\frac{1}{2f} \right)^{\frac{1}{3}} - 1 \right],\tag{8}$$

where G_m = 2.64 GPa (shear modulus of the matrix), b = 0.32 nm [45,46], λ = 230.56 nm (the effective planar interparticle spacing), f = 0.00283 is the volume fraction of reinforcements calculated from the weight fraction and d_p is the average diameter of the refinement particles from Table 2. The calculated Orowan strengthening mechanism is listed in Table 8.



Figure 13. Orowan mechanism of moving the location line from zone(a)-(c) and Orowan loop formation.

The CTE, the shear transfer effect of the load and Orowan strengthening development in the composites are presented in Table 8. All the effect values (increasing trend of total cumulative) show that the compression test delays crack propagation, dramatically strengthens the interface of the alloys and increases the compression properties (increasing trend), as shown in Table 5 [17].

4.3. Quantitative Phases Influences on Mechanical Properties

An outstanding amount of secondary phases was scattered throughout alloy code 2, and the existence of SiCp increased its microhardness [12]. A refined grain structure means that β -SiCp can restrict grain growth and maintain excellent dispersion (Figure 11b); thus, SiCp is vital for accomplishing excellent mechanical properties, such as tensile and impact properties [33,34].

However, the tensile and impact properties of alloy codes 3 and 4 were reduced in the presence of SiCp, Mg_2Si and C_{60} , which can cause microcracks that coalescence into large cracks [12], as shown in Figure 12c.

Nevertheless, the addition of 1 wt.% β -SiCp (alloy code-3) substantially decreased the mechanical strength (the yield and ultimate properties) because of the sinking and floating comportment of SiCp compared with AZ91 alloy because of its superior density and surface tension. The main prerequisite for these mechanical properties changed because of the presence of new intermetallic Al and Mn phases and massive Mg₁₇Al₁₂ (Figure 10c–d) phases. Figure 10c specifies the EDS results for alloy code-3. Alloy code 3 has several combinations of Al (48.78 wt.%) and Mn (47.26 wt.%), which are considered intermediate brittle phases, such as Al₆Mn and others presented in Tables 7 and 9 in the massive Mg₆Al₁₂ phase. Similarly, Figure 10d shows an enormous solid phase of Mg (52.19 wt.%) and Al (47.76 wt.%). A massive discontinuous Mg₆Al₁₂ phase appeared when the β -SiCp amount increased to 1 wt.%. Moreover, XRD studies also confirmed the existence of these phases, which dominated the mechanical strength of alloy code 3. In addition, previous similar studies have demonstrated that Al–Mn, β -Mg₁₇Al₁₂ and Mg₂Si diminish mechanical properties [12,34].

Al–Mn Phases	Alloy Code 1	Alloy Code 2	Alloy Code 3	Alloy Code 4
Al ₇₈ Mn ₂₂	1	×	×	1
Al _{0.43} Mn _{0.47}	1	×	1	1
Al ₆ Mn [51]	1	×	×	✓
Al ₈₀ Mn ₂₀	×	×	×	1
Al ₁₀ Mn ₃	×	×	×	✓
Al ₈₆ Mn ₁₄	×	×	1	×
Al ₈₁ Mn ₁₉	×	1	1	1
Al ₇₇ Mn ₂₃	1	1	×	×

Table 9. Combinations of Al–Mn phases present in alloy codes 1 to 4 based on XRD textures. ([\checkmark] and [\times] denote the presence and absence of the respective phase, respectively).

In the impact test, alloy code 4 had multiple Al–Mn-constructed brittle phases, as shown in Tables 7 and 9, based on the XRD results. The mechanical properties were remarkably reduced because of these Al–Mn-based materials [51]. The results of the tensile test of alloy code 4 displayed in Figure 12d show that these elements can produce substantial amounts of brittle heterogeneous phases of C_{60} , SiCp and Mg₂Si, which can reduce mechanical properties slightly compared to those of alloy code 2 because of the formation of small microcracks (Figure 14d).



Figure 14. Fractography of the compression-ruptured surfaces of (**a**) pure AZ91 matrix, (**b**) AZ91 + 0.5 wt.% SiCp, (**c**) AZ91 + 1 wt.% SiCp and (**d**) AZ91 + 0.5 wt.% SiCp + 1 wt.% Bi + 0.4 wt.% Sb.

4.4. Fracture Surface Analysis

As depicted in Figures 13–15, the fracture morphology of pure AZ91 and reinforced AZ91 composites (codes 1 to 4) was studied by SEM after tensile, compression and impact tests. The studies reflect the occurrence of a combination of ductile and brittle fractures. β -Mg₁₇Al₁₂ obstructed the commencement of cracks and deformation in the AZ91 matrix [52].



Figure 15. Fractography of the impact-ruptured surfaces of (**a**) pure AZ91 matrix, (**b**) AZ91 + 0.5 wt.% SiCp, (**c**) AZ91 + 1 wt.% SiCp and (**d**) AZ91 + 0.5 wt.% SiCp + 1 wt.% Bi + 0.4 wt.% Sb.

The bunting cracks, microcracks and cavities detected in the 1 wt.% SiCp-reinforcementadded composite alloy (code 3) shown in Figure 12c were more than those in alloy codes 1 and 2 in Figure 12a,b, respectively. Voids are inevitable in the stir casting method with environs but can be governable. These voids exhaust the gas induced in the contiguous particles and augment viscosity. The creation of voids and a secondary phase formulates the stress concentration between particles and matrix. Furthermore, the large number of particles leads to a higher concentration owing to the higher dislocation density [17]. When the load increases during tensile tests, the cleavage facet transfers and breaks the intrinsically brittle structure in the transverse direction of the plane [52]. The load can be moved from the α -Mg matrix to the reinforcement particles; at this moment, an enormous crack is initiated by the reinforcement particles [11]. The tensile values of alloy code 4 decrease slightly because of the reinforcement increments, which can formulate a large number of microcracks. In addition, the presence of Al–Mn-based components (Table 9) can substantially reduce mechanical strength [51].

The number of shear bands (shear tongues) that exist in the fractured surface increased due to the cumulative nano-and micro-reinforcements [11] in alloy codes 2 to 4 compared with alloy code 1, as shown in Figure 14. The cavities and dimples in the fracture surface

decreased from alloy codes 2 to 4. In addition, the value of the strengthening mechanism had an increasing trend, as shown in Table 8. The cohesion force between matrix and refinement increased and dimples reduced because of grain refinement. In addition, the interface between plastic flow was due to the rigid reinforcements in the AZ91 matrix [17].

In alloy code 2, β -Mg₁₇Al₁₂ diffused along the grain boundary increased the impact property dramatically. The presence of Mg₂Si in alloy code 3 can initiate the formation of micro- and nano-cracks [12]. However, the impact value of alloy code 4 (AZ91 + 0.5 wt.% SiCp + 1 wt.% Bi + 0.4 wt.% Sb) was lower than that of as-cast AZ91 because of the number of reinforcements whose crystal structures existed in the matrix.

The twin boundary fracture, microcracks and surface cracks in the impact test fracture morphology are shown in Figure 15. Alloy code 4 had huge dimples, microcracks and surface cracks compared with the other alloy codes, as shown in Figure 15d. The assortment of C_{60} , SiCp and Mg₂Si heterogeneous phases can reduce the impact strength of the materials, and the presence of huge Al₆Mn phases also plays a vital role in reducing the strength of the impact values [51]. The HCP structures of twin boundary cracks were observed in alloy code 4, and these major boundary cracks, which lead to the reduction of energy values, are shown in Figure 15d.

5. Conclusions

The different amalgamations of the hybrid nanoelement (β -SiCp) and microelements (Bi and Sb) of AZ91 (alloy codes 1 to 4) MMCs were investigated. Microstructure evaluation, phase quantitative analysis, the strengthening mechanism (CTE), the shear transfer effect and the Orowan effect and their impact on mechanical properties (tensile, compression and impact) were studied in detail. The following conclusions are derived from the present study:

- The addition of β -SiCp (0.5 and 1 wt.%) nanoparticles resulted in grain refinement, particle strengthening and creation of quantitative phases Mg₂Si (cubic) and SiC (rhombo.h.axes), which improved the microhardness of the AZ91 matrix by 18.6% and 14.66%, respectively. Similarly, a 17.49% increment in microhardness was generated by adding 0.5% SiCp + 1% Bi + 0.4% Sb to the AZ91 matrix. However, the microhardness of alloy codes 3 and 4 slightly decreased because of the reduction in Mg_{0.97}Zn_{0.03} (hexagonal) brittle phases.
- The small number of 0.5 wt.% β-SiC nanoparticles remarkably improved the tensile and yield values by 40.10% and 5.47% (169.33 and 72.82 MPa, respectively). The best yield strength of 82.75 MPa (increased by 19.85%) was obtained from the AZ91 matrix (alloy code 4) with 0.5 wt.% SiCp, 1 wt.% Bi and 0.4 wt.% Sb as reinforcements. Strength was related to the mismatch of the CTE and the existence of quantitative phases of Mg₂Si, Mg₃Bi₂, Mg₃Sb₂ and Mg_{0.97}Zn_{0.03} in the AZ91 matrix.
- Compression properties increased by 2.68%, 6.23% and 8.38% for alloy codes 2 to 4, respectively. CTE augmentation; the shear transfer effect of the load; the Orowan strengthening effect and the dispersion of quantitative phases Mg₂Si, Mg₃Bi and Mg₃Sb₂ in the composites presented delays in crack propagation and occurrence at the particle–matrix interface.
- The addition of β-SiC remarkably reduced brittle Al–Mn-based phases; thus, the absorbed Charpy impact energy improved by 236% (2.89 J) and 192% (2.35 J) for alloy codes 2 and 4, respectively.
- Alloy code 4 (0.5 wt.% SiCp + 1 wt.% Bi + 0.4 wt.% Sb) gained impact energy negatively, 0.98 J (-80%), because of the increment in reinforcement quantity and the creation of C₆₀, SiCp and Mg₂Si phases. Al–Mn-based brittle phases, such as Al₆Mn, were observed in the XRD pattern. A large number of dimples and microcracks were created in alloy code 4's impact test fracture morphology because of the sinking and floating comportments of SiCp, Sb and Bi.

Particle debonding, the coalescence of voids and the nucleation growth of ductile and brittle fractures were detected in tensile morphology. Debonding of microcracks was perceived in the impact of the fragile fracture surface.

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References

- 1. Yang, Z.R.; Wang, S.Q.; Gao, M.J.; Zhao, Y.T.; Chen, K.M.; Cui, X.H. A new-developed magnesium matrix composite by reactive sintering. *Compos. Part A Appl. Sci. Manuf.* 2008, *39*, 1427–1432. [CrossRef]
- Meng, F.; Lv, S.; Yang, Q.; Qin, P.; Zhang, J.; Guan, K.; Huang, Y.; Hort, N.; Li, B.; Liu, X.; et al. Developing a die casting magnesium alloy with excellent mechanical performance by controlling intermetallic phase. *J. Alloys Compd.* 2019, 795, 436–445. [CrossRef]
- 3. Joost, W.J.; Krajewski, P.E. Towards magnesium alloys for high-volume automotive applications. *Scr. Mater.* **2017**, *128*, 107–112. [CrossRef]
- 4. Jin, T.; Zhou, Z.; Qiu, J.; Wang, Z.; Zhao, D.; Shu, X.; Yan, S. Investigation on the yield behavior of AZ91 magnesium alloy. J. Alloys Compd. 2018, 738, 79–88. [CrossRef]
- Lin, X.Z.; Chen, D.L. Strain controlled cyclic deformation behavior of an extruded magnesium alloy. *Mater. Sci. Eng. A* 2008, 496, 106–113. [CrossRef]
- 6. Lee, S.W.; Park, S.H. Static recrystallization mechanism in cold-rolled magnesium alloy with off-basal texture based on quasi in situ EBSD observations. *J. Alloys Compd.* **2020**, *844*, 156185. [CrossRef]
- Martynenko, N.; Lukyanova, E.; Serebryany, V.; Prosvirnin, D.; Terentiev, V.; Raab, G.; Dobatkin, S.; Estrin, Y. Effect of equal channel angular pressing on structure, texture, mechanical and in-service properties of a biodegradable magnesium alloy. *Mater. Lett.* 2019, 238, 218–221. [CrossRef]
- Mironov, S.; Onuma, T.; Sato, Y.S.; Kokawa, H. Microstructure evolution during friction-stir welding of AZ31 magnesium alloy. *Acta Mater.* 2015, 100, 301–312. [CrossRef]
- 9. Bian, M.; Huang, X.; Chino, Y. Improving flame resistance and mechanical properties of magnesium–silver–calcium sheet alloys by optimization of calcium content. *J. Alloys Compd.* **2020**, *837*, 155551. [CrossRef]
- Wang, H.Y.; Rong, J.; Yu, Z.Y.; Zha, M.; Wang, C.; Yang, Z.Z.; Bu, R.Y.; Jiang, Q.C. Tensile properties, texture evolutions and deformation anisotropy of as-extruded Mg-6Zn-1Zr magnesium alloy at room and elevated temperatures. *Mater. Sci. Eng. A* 2017, 697, 149–157. [CrossRef]
- 11. Yang, C.; Zhang, B.; Zhao, D.; Wang, X.; Sun, Y.; Xu, X.; Liu, F. Microstructure evolution of as-cast AlN/AZ91 composites and room temperature compressive properties. *J. Alloys Compd.* **2019**, 774, 573–580. [CrossRef]
- 12. Huang, S.J.; Ali, A.N. Effects of heat treatment on the microstructure and microplastic deformation behavior of SiC particles reinforced AZ61 magnesium metal matrix composite. *Mater. Sci. Eng. A* 2018, *711*, 670–682. [CrossRef]
- Nie, K.; Guo, Y.; Deng, K.; Kang, X. High strength TiCp/Mg-Zn-Ca magnesium matrix nanocomposites with improved formability at low temperature. J. Alloys Compd. 2019, 792, 267–278. [CrossRef]
- Sun, X.F.; Wang, C.J.; Deng, K.K.; Nie, K.B.; Zhang, X.C.; Xiao, X.Y. High strength SiCp/AZ91 composite assisted by dynamic precipitated Mg17Al12 phase. J. Alloys Compd. 2018, 732, 328–335. [CrossRef]
- 15. Xiao, P.; Gao, Y.; Yang, X.; Xu, F.; Yang, C.; Li, B.; Li, Y.; Liu, Z.; Zheng, Q. Processing, microstructure and ageing behavior of in-situ submicron TiB2 particles reinforced AZ91 Mg matrix composites. *J. Alloys Compd.* **2018**, *764*, 96–106. [CrossRef]
- 16. Wang, X.J.; Hu, X.S.; Liu, W.Q.; Du, J.F.; Wu, K.; Huang, Y.D.; Zheng, M.Y. Ageing behavior of as-cast SiCp/AZ91 Mg matrix composites. *Mater. Sci. Eng. A* 2017, 682, 491–500. [CrossRef]
- 17. Huang, S.J.; Abbas, A. Effects of tungsten disulfide on microstructure and mechanical properties of AZ91 magnesium alloy manufactured by stir casting. *J. Alloys Compd.* **2020**, *817*, 153321. [CrossRef]
- 18. Xiao, P.; Gao, Y.; Xu, F.; Yang, C.; Li, Y.; Liu, Z.; Zheng, Q. Tribological behavior of in-situ nanosized TiB2 particles reinforced AZ91 matrix composite. *Tribol. Int.* 2018, 128, 130–139. [CrossRef]
- 19. Ganguly, S.; Mondal, A.K. Influence of SiC nanoparticles addition on microstructure and creep behavior of squeeze-cast AZ91-Ca-Sb magnesium alloy. *Mater. Sci. Eng. A* 2018, 718, 377–389. [CrossRef]

- 20. Zhu, Y.P.; Jin, P.P.; Fei, W.D.; Xu, S.C.; Wang, J.H. Effects of Mg2B2O5 whiskers on microstructure and mechanical properties of AZ31B magnesium matrix composites. *Mater. Sci. Eng. A* 2017, *684*, 205–212. [CrossRef]
- Sun, X.F.; Wang, C.J.; Deng, K.K.; Kang, J.W.; Bai, Y.; Nie, K.B.; Shang, S.J. Aging behavior of AZ91 matrix influenced by 5 μm SiCp: Investigation on the microstructure and mechanical properties. *J. Alloys Compd.* 2017, 727, 1263–1272. [CrossRef]
- Elen, L.; Zengin, H.; Turen, Y.; Turan, M.E.; Sun, Y.; Ahlatci, H. Effects of bismuth (bi) additions on microstructure and mechanical properties of AZ91 alloy. In Proceedings of the Metal 2015—24th International Conference on Metallurgy and Materials, Conference Proceedings, Brno, Czech Republic, 3–5 June 2015; pp. 1124–1128.
- 23. Guangyin, Y.; Yangshan, S.; Wenjiang, D. Effect of bismuth and antimony additions on the microstructure and mechanical properties of AZ91 magnesium alloy. *Mater. Sci. Eng. A* 2001, *308*, 38–44. [CrossRef]
- 24. Abbas, A.; Huang, S.J. Investigation of severe plastic deformation effects on microstructure and mechanical properties of WS2/AZ91 magnesium metal matrix composites. *Mater. Sci. Eng. A* 2020, 780, 139211. [CrossRef]
- 25. ASTM International. E3 Preparation of Metallographic Specimens. Annu. B ASTM Stand. 2017, 11, 1–17.
- 26. ASTM E8 ASTM E8/E8M standard test methods for tension testing of metallic materials 1. *Annu. B. ASTM Stand.* 4 2010, 1–27. [CrossRef]
- 27. E9-19 Standard Test Methods of Compression Testing of Metallic Materials at Room Temperature. *ASTM B. Stand.* **2019**, 1–10. [CrossRef]
- 28. Yi, Y.S.; Li, R.F.; Xie, Z.Y.; Meng, Y.; Sugiyama, S. Effects of reheating temperature and isothermal holding time on the morphology and thixo-formability of SiC particles reinforced AZ91 magnesium matrix composite. *Vacuum* **2018**, *154*, 177–185. [CrossRef]
- ASTM International. ASTM E23—18, Standard Test Methods for Notched Bar Impact Testing of Metallic Materials. ASTM Int. 2018, 1–26. [CrossRef]
- Qiao, X.G.; Ying, T.; Zheng, M.Y.; Wei, E.D.; Wu, K.; Hu, X.S.; Gan, W.M.; Brokmeier, H.G.; Golovin, I.S. Microstructure evolution and mechanical properties of nano-SiCp/AZ91 composite processed by extrusion and equal channel angular pressing (ECAP). *Mater. Charact.* 2016, 121, 222–230. [CrossRef]
- Viswanath, A.; Dieringa, H.; Ajith Kumar, K.K.; Pillai, U.T.S.; Pai, B.C. Investigation on mechanical properties and creep behavior of stir cast AZ91-SiCp composites. J. Magnesuim Alloy. 2015, 3, 16–22. [CrossRef]
- 32. Srinivasan, A.; Swaminathan, J.; Pillai, U.T.S.; Guguloth, K.; Pai, B.C. Effect of combined addition of Si and Sb on the microstructure and creep properties of AZ91 magnesium alloy. *Mater. Sci. Eng. A* 2008, 485, 86–91. [CrossRef]
- 33. Zhou, X.; Su, D.; Wu, C.; Liu, L. Tensile mechanical properties and strengthening mechanism of hybrid carbon nanotube and silicon carbide nanoparticle-reinforced magnesium alloy composites. *J. Nanomater.* **2012**, 2012, 851862. [CrossRef]
- Zhao, W.; Huang, S.J.; Wu, Y.J.; Kang, C.W. Particle size and particle percentage effect of AZ61/SiCp magnesium matrix microand nano-composites on their mechanical properties due to extrusion and subsequent annealing. *Metals* 2017, 7, 293. [CrossRef]
- 35. Srinivasan, A.; Pillai, U.T.S.; Pai, B.C. Effects of elemental additions (Si and Sb) on the ageing behavior of AZ91 magnesium alloy. *Mater. Sci. Eng. A* **2010**, 527, 6543–6550. [CrossRef]
- Guangyin, Y.; Yangshan, S.; Weiming, Z. Improvements of tensile strength and creep resistance of Mg-9Al based alloy with antimony addition. J. Mater. Sci. Lett. 1999, 18, 2055–2057. [CrossRef]
- 37. Wang, Q.; Chen, W.; Ding, W.; Zhu, Y.; Mabuchi, M. Effect of Sb on the Microstructure and Mechanical Properties of AZ91 Magnesium Alloy. *Metall. Mater. Trans. A* 2001, *32*, 787–794. [CrossRef]
- 38. Bag, A.; Zhou, W. Tensile and fatigue behavior of AZ91D magnesium alloy. J. Mater. Sci. Lett. 2001, 20, 457–459. [CrossRef]
- 39. Srinivasan, A.; Swaminathan, J.; Gunjan, M.K.; Pillai, U.T.S.; Pai, B.C. Effect of intermetallic phases on the creep behavior of AZ91 magnesium alloy. *Mater. Sci. Eng. A* 2010, 527, 1395–1403. [CrossRef]
- 40. Srinivasan, A.; Pillai, U.T.S.; Pai, B.C. Microstructure and mechanical properties of Si and Sb added AZ91 magnesium alloy. *Metall. Mater. Trans. A Phys. Metall. Mater. Sci.* 2005, *36*, 2235–2243. [CrossRef]
- 41. Nguyen, Q.B.; Gupta, M. Increasing significantly the failure strain and work of fracture of solidification processed AZ31B using nano-Al2O3 particulates. *J. Alloys Compd.* 2008, 459, 244–250. [CrossRef]
- 42. Inegbenebor, A.O.; Bolu, C.A.; Babalola, P.O.; Inegbenebor, A.I.; Fayomi, O.S.I. Aluminum Silicon Carbide Particulate Metal Matrix Composite Development Via Stir Casting Processing. *Silicon* **2018**, *10*, 343–347. [CrossRef]
- 43. Xiao, P.; Gao, Y.; Xu, F.; Yang, S.; Li, B.; Li, Y.; Huang, Z.; Zheng, Q. An investigation on grain refinement mechanism of TiB2 particulate reinforced AZ91 composites and its effect on mechanical properties. J. Alloys Compd. 2019, 780, 237–244. [CrossRef]
- 44. Aydin, F.; Sun, Y.; Emre Turan, M. The Effect of TiB2 Content on Wear and Mechanical Behavior of AZ91 Magnesium Matrix Composites Produced by Powder Metallurgy. *Powder Metall. Met. Ceram.* **2019**, *57*, 564–572. [CrossRef]
- 45. Deng, K.; Shi, J.; Wang, C.; Wang, X.; Wu, Y.; Nie, K.; Wu, K. Microstructure and strengthening mechanism of bimodal size particle reinforced magnesium matrix composite. *Compos. Part A Appl. Sci. Manuf.* **2012**, *43*, 1280–1284. [CrossRef]
- 46. Habibnejad-Korayem, M.; Mahmudi, R.; Poole, W.J. Enhanced properties of Mg-based nano-composites reinforced with Al₂O₃ nano-particles. *Mater. Sci. Eng. A* **2009**, *519*, 198–203. [CrossRef]
- Huang, S.J.; Lin, P.C. Grain refinement of AM60/Al2O3p magnesium metal-matrix composites processed by ECAE. *Kov. Mater.* 2013, 51, 357–366.
- 48. Karbalaei Akbari, M.; Baharvandi, H.R.; Shirvanimoghaddam, K. Tensile and fracture behavior of nano/micro TiB2 particle reinforced casting A356 aluminum alloy composites. *Mater. Des.* **2015**, *66*, 150–161. [CrossRef]

- 49. Huang, S.J.; Ho, C.H.; Feldman, Y.; Tenne, R. Advanced AZ31 Mg alloy composites reinforced by WS2 nanotubes. *J. Alloys Compd.* **2016**, *654*, 15–22. [CrossRef]
- 50. Saba, F.; Zhang, F.; Liu, S.; Liu, T. Reinforcement size dependence of mechanical properties and strengthening mechanisms in diamond reinforced titanium metal matrix composites. *Compos. Part B Eng.* **2019**, *167*, 7–19. [CrossRef]
- Tokarski, T. Effect of rapid solidification on the structure and mechanical properties of AZ91 magnesium alloy. *Solid State Phenom.* 2012, 186, 120–123. [CrossRef]
- 52. Abbas, A.; Huang, S.J. ECAP effects on microstructure and mechanical behavior of annealed WS2/AZ91 metal matrix composite. *J. Alloys Compd.* **2020**, *835*, 155466. [CrossRef]