THE MICROSTRUCTURE AND MECHANICAL PROPERTIES OF HOT ROLLED AT31 MG ALLOYS MODIFIED BY LA AND GD

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I certify that in my opinion the thesis submitted by Imran Ali titled “THE MICROSTRUCTURE AND MECHANICAL PROPERTIES OF HOT ROLLED AT31 MG ALLOYS MODIFIED BY LA AND GD” is fully adequate in scope and in quality as a thesis for the degree of Master of Science.

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This thesis is accepted by the examining committee with a unanimous vote in the Department of Metallurgical and Materials Engineering as a Master of Science thesis. Jan 03, 2022

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"I declare that all the information within this thesis has been gathered and presented in accordance with academic regulations and ethical principles and I have according to the requirements of these regulations and principles cited all those which do not originate in this work as well."

Imran Ali

ABSTRACT

M. Sc. Thesis

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The manufacturers of this age are facing a lot of troubles in producing the materials possessing light weight and best amount of efficiency. For this purpose, magnesium is the perfect choice for the producers for replacing the materials such as steel and alloys containing aluminum and plastic materials. In this work, AT31 Mg alloy was used as major materials. After casting, homogenization heat treatment was applied at 350 degrees centigrade for 12 hours respectively. Mg-2.5Al-1.0Sn-0.3Mn-0.4La-0.16,0.66,1.33Gd alloys were produced through hot rolling process. AT31 Mg alloy with different amount Gd addition, underwent 20% deformation per pass at 1.5 m/min, 4.7 m/min and 10 m/min rolling speeds. Total number of passes is 8 for each specimen. Optical microscopy and scanning electron microscopy were done upon the rolled, grinded, polished, and etched specimen. In optical microscopy, coarse grains, fine grains, and twinning were noticed while in scanning electron microscopy, widely dispersed polygonal and spherical shaped secondary phases were noticed.

Average grain size, twin fraction, Brinell hardness were found to be decreased with increased rolling speed, but ultimate tensile strength (UTS) increased by increasing rolling speed. Cleavage plane and dimples were found in fracture surface analysis and the intensity of Mg peaks was identified in XRD results.

Key Words : Mg alloys, Al alloys, rare earth metals, Hot rolling.
Science Code : 91519

ÖZET

Yüksek Lisans Tezi

LA VE GD İLAVELİ SICAK HADDELENMİŞ AT31 MG ALAŞIMLARININ MİKRO YAPISI VE MEKANİK ÖZELLİKLERİ

Imran ALI

Karabük Üniversitesi
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Metalürji ve Malzeme Mühendisliği Anabilim Dalı

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Bu çağın üreticileri, hafifliği ve verimi en iyi olan malzemeleri üretmekte pek çok sıkıntıyla karşı karşıyadır. Bu amaçla, çelik ve alaşımları, alüminyum ve plastik malzemelerin yerine magnezyum, üreticiler için mükemmel bir seçimdir. Bu çalışmada ana malzeme olarak AT31 Mg alaşımı kullanılmıştır. Döküm işleminden sonra 350 santigrat derecede 12 saat homojenizasyon ısıl işlemi uygulanmıştır. Mg-2.5Al-1.0Sn-0.3Mn-0.4La-0.16,0.66,1.33Gd alaşımları sıcak haddeleme işlemi ile üretilmiştir. Farklı miktarda Gd ilaveli AT31 Mg alaşımı, 1.5 m/dk, 4.7 m/dk ve 10 m/dk haddeleme hızlarında paso başına %20 deformasyona uğramıştır. Her numune için toplam paso sayısı 8'dir. Haddelenmiş, zımparalanmış, parlatılmış ve dağlanan numune üzerinde optik mikroskop ve taramalı elektron mikroskobu incelemesi yapılmıştır. Optik mikroskopta iri taneler, ince taneler ve ikizlenme görülürken, taramalı elektron mikroskobunda geniş çapta dağılmış poligonal ve küresel şekilli ikincil fazlar fark edilmiştir. Ortalama tane boyutu, ikizlenme fraksiyonu, Brinell sertliğinin haddeleme hızının artmasıyla azaldığı, ancak nihai çekme mukavemetinin haddeleme hızının artmasıyla arttığı bulundu. Kırık yüzey analizinde kopma düzlemi ve şeritler bulundu ve XRD sonuçlarında Mg piklerinin yoğunluğu belirlendi.

Anahtar Kelimeler : AZ31B, Nd, La, korozyon, Mekanik Perçinleme.
Bilim Kodu : 91519

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PART 1

INTRODUCTION

1.1. MAGNESIUM

The fabricators working on production in this era are supposed to bear a lot of circumstances while producing the materials containing light weight and more efficiency. Magnesium is best option for developers regarding production and manufacturing and can be used instead of materials like steel, alloys containing aluminum and plastic. In recent times, there was very less research and developmental work being done upon magnesium because the reason was its high price. Nowadays, magnesium has been taken under consideration because the price of magnesium alloys has been decreased as compared to past. The sturdy properties of magnesium alloys have been preferred most to other metals like copper and aluminum-based alloys [1]. Casting is being preferred most to produce magnesium-based alloys and shows about 98% structural applications of it [2]. Magnesium alloys are widely used in automotive, aerospace, and medical fields due to their lower density, fine specific strength, and good damping capacity and magnesium alloys possess less weight when we compare to other alloys like aluminum alloys also strength-to-weight ratio of magnesium alloys is very good and material producers and engineers are very thankful of magnesium alloys [3]. In the field of automotive, magnesium alloys play very major role because of their high stiffness, high capacity to absorb vibrations and supreme cutting capability [4].

1.2. ALLOYING ELEMENTS

1.2.1. Aluminum

Aluminum is well known due to its some major properties like it has superior ductility and formability and it has very good resistance to corrosion, and conduction of electricity and heat [5]. Aluminum is usually extracted from mineral bauxite and then using a process called Bayer Process is transformed to aluminum oxide commonly knowns as alumina. Total of the alumina consumed by the market economy aluminum industry is processed using Bayer Process which hasn't been changed since it was initiated in Germany by Karl Josef Bayer in the year 1988 [6,7]. Through Hall-Heroult process, most of the aluminum production is done whose pre features remained unchanged since copyrights were provided in 1886, in America by Charles M. Hall and in France by Paul L.T. Heroult [6,7].

1.2.2. Zinc

Zinc comes at the 4^{th} number following the metals iron, aluminum, and copper. The supply of the zinc in the world has been increased to 13.4 Mt as the demand was 13.77 Mt [8]. The handsome amount of zinc has been made usable by recycling process and estimation of its production is about 20% to 40% for the whole world [9]. About one half of the total zinc is used in galvanizing steel and prevention from corrosion [10]. Freshly, it has been discovered that zinc can be a good biodegradable metal and can easily be implemented at the places of iron and magnesium [11]. Approximately, 15% from the total amount of the zinc present in the world is treated like a base metal and zinc-based alloys are produced of it [12]. Zinc alloys can easily be found in the market in the form of wrought alloys, drawn into wires, extrusion applied and forged items. Recently, a latest zinc alloy for extrusion and forging has been fabricated and available in the market [13].

1.2.3. Tin

Tin possesses allotropy at normal and metallic forms and is body centered tetragonal at the temperature which is above transformation which is about 13.2 degree centigrade [14]. Gray tin acts like a knob of materials having property of friability on the metal form of it and seems to be a thing made of corrosion, hence, its modification is not different as compared to corrosion [15]. The modification process was carried through high-resolution scanning electron microscopy to get to know about adaptation relationship among the two allotropic materials [16].

1.2.4. Manganese

Glass and ceramic producers of ancient times have brought manganese in their production but in 18th century, the manufacturers of iron and steel got attracted to manganese for steel production [17]. South Africa has been regarded as world's number one manganese producer because it produces about 82% of the total manganese in the world [17]. Steels containing manganese offer many benefits like cheap cost and are good resistant to corrosion [18].

1.3. RARE EARTH ELEMENTS

1.3.1 Gadolinium

Gadolinium has been carried in use by most of the material researchers in search of better properties for different alloys. It has been acknowledged that the creep and corrosion resisting properties of Mg alloys have been improved by addition of some rare earth elements like Ce, La, Gd, Nd to it [19]. The rare earth metals are the reason for forming the reinforcing intermetallic phase which possesses great stability even at high temperature and the rare earth elements possessing greater solubility are responsible for forming a solution in α-Mg matrix which is in solid form and provide great sturdy properties to Mg alloys [20].

1.3.2. Lanthanum

Rare earth elements are mostly used improve the existing properties of different alloys. The inclusion of rare earth elements like Lanthanum to Mg, Al and different type of alloys mostly provides improvement in the alloys like it increases sturdiness and decreases the weight of the alloy and make it very light weight which is very useful for manufacturing the materials [21]. Rare earth elements provide great yield strength to Mg alloys [22].

1.4. HOT DEFORMATION METHODS

1.4.1. Hot Rolling

Hot rolling is a deformation process which is used to reduce the thickness of metal while metal is being passed through a pair of rolls. Hot rolling is used to make the thickness of the specimen uniform and give the required and valuable properties to the material being rolled between the rolls. The fabrication of materials using hot rolling has been a major trouble and most of the work has been done upon it in the recent times also the artificial intelligence methods are taken in use for it in both ways theoretically and practically [23].

1.4.2. Extrusion

It is a mechanism of forming the things of measured cross-sectional area by passing the material across the die of required cross section. We have two important advantages of it and number one is that it can create very complex cross sections and second one is that it can be successfully applied on ductile materials because the materials face stresses like compressive stress and shear stress. Hot melt extrusion has been used as most prominent technology in the industries especially in plastic industries for last 70 years approximately but in pharmaceutical companies it has been used as a substitute for various solid applications. More than nine dozen of research publications have been made available by the publishers regarding it [24]. Recently, FRC elements production has been done by using extrusion process [25].

1.5. APPLICATIONS OF MG SHEET MATERIALS

The applications of Mg sheet materials play very important role to get reduction in different ways like protection from dents in vehicles. The lightweight requirement for different parts in different industries particularly in automotive industry has got the attention of researchers to work upon the Mg-Al alloys to get the appropriate applications such as low consumptions of fuel and decrease the leakages in vehicles [26]. Relative to this type of work the research and working upon the alloys within series of 6xxx has been increased gradually within the recent years [27].

PART 2

LITERATURE REVIEW

As we are having a deep look upon the past time, a great amount of work has been done upon AZ91 alloy because of its properties such as great sturdiness and excellent resistance to corrosion and castability. AZ91 is a commercialized Mg alloys which doesn't contain any of the rare earth elements and has been used in different industries especially, automotive and aerospace [28]. It has been noticed that the high content of Al in the alloy is responsible for producing coarse reticular particles of $Mg_{17}Al_{12}$ during the material is carried under casting mechanism and these reticular particles usually effect the mechanical properties of it in negative way and can provide different faults and worsen the mechanical properties of the material [29]. For increasing the microstructure and mechanical properties of the alloy it has been passed through a homogeneous treatment before doing any heat mechanical process under high temperature, just taking an example of $Mg_{17}Al_{12}$ which was heated about at 415 degrees centigrade for 24 hours almost just to minimize the negative effect of casting upon it [30]. The rare earth metals called Nd, Ce and Y were added to the magnesium sheets to achieve weak texture, the amount of the added rare earth metals to get weak texture is directly concerned with solvability of the material in solid form to which that amount of rare earth metals is added [31]. The debilitating of texture leads the material to have such a good formability and have concern with the deformation bands carrying twins and diminution of the growth of the grain and the spotted deformation band carrying twins with them were found different from each other in macroscopic shear bands which were due to some inhomogeneous deformation and the latter bands bends were noticed to be the cause of formation of cracks while sheets were passed through sheet-forming process and that was the reason for decrement of formability in the sheets [31]. After the inclusion of rare earth metals to the ZEK100 Mg alloys, the alloy was carried through hot rolling and another process called annealing at room temperature just to know some properties of the sheet like formability and anisotropy and separate effect of each rare earth element on these properties and we became able to know that each rare earth metal was responsible for creating different microstructures and consisting some alterations in the average of grain size and solvability in solid form and provided it distinct properties [32]. In the presence of less yield strength which is due to the rare earth elements textures, the sheets were able to have ultimate tensile strength (UTS) which is about 230MPa to 240MPa approximately which is almost equal to that of commercialized AZ31 sheet and those sheets containing rare earth metals were found have isotropic *r*-values which are almost equal to that which were found from strains having same width and thickness as well [32]. The sheet processed by hot rolling from Mg-Gd-Y-Zn-Zr alloy containing high strength and the twins got almost vanished after doing grievous hot rolling and the microstructure also got to be recrystallized completely and the texture of the base of the rolled sheets with base of the plane was found parallel to the plane of rolling and the added rare earth elements and application of recrystallization were responsible for weakening the texture intensity of the sheet [33]. It was noticed that a type of LPSO phase called 18R was laying on the boundaries of the grain and another sort known as 14R was noticed scattered interiorly and the Mg sheet after applying hot rolling upon it was reduced about 96% and found to have supreme mechanical properties and the ultimate tensile strength (UTS) was about 403MPa and the tensile yield strength (TYS) was about 318MPa and elongation to failure was 13.7% and by increasing the reduction in rolling, the properties like anisotropy's sturdiness and elongation were seen to be increased [33]. Different types of rare earth elements were combined within the Mg alloys and all of them showed different properties like Mg-Y alloy was noticed to possess handsome sturdiness and at the same time Mg-Er alloys were carrying supreme ductility while Mg-Zn-RE ternary alloy was found carry both properties and supreme tensile strength as well [34].

2.1. THE EFFECT OF SN ON THE MICROSTRUCTURE AND MECHANICAL PROPERTIES OF MG-AL BINARY ALLOYS

By applying the solution heat treatment and ageing of both types i.e., artificially, and directly and according to the condition of as-cast, the heat treatments of the types T5 and T6 were applied, and it was investigated that the microstructure remained unstable at the time of treating the Mg-Al alloy containing Sn at the increased temperature and in the process, β phase occurs in Mg_2Sn and ternary alloys and at the elevated melting temperature Mg_2Sn influenced the process of precipitation also Mg-Sn alloy was found to have best creep and resistance to corrosion [35]. Sn was included to AZ91 alloy and most of the results were found by adding Sn to AZ91 such as lamellar eutectics were seen to be converted into completely isolated β eutectics and congregated phase found within β eutectics in the weight of 2% and resulted in the increment of fluidity of AZ91 by 0.5 wt.% but the fluidity gets reduced when the amount of Sn crosses the limit of 0.5 wt.% and hot tear susceptibility (HTS) was caught decreasing by adding 0.5 wt.% of Sn and UTS, YS, and ER were caught increasing by the addition of Sn content more than that of 0.5 wt.% [36].

2.2. THE EFFECT OF MN ON THE MICROSTRUCTURE AND MECHANICAL PROPERTIES OF MG-AL BINARY ALLOYS

The efficiency of refining of the grain and refining of mechanism of the manganese carbonate together with Al containing magnesium alloy were being studied and manganese carbonate was noticed to have a good amount of efficiency of refining in Mg-9 wt.% Al alloys, and a decrement about 61 μm was noticed in the average size of the grain by including 0.6 wt.% of $MnCO_3$ [37]. The immunization of manganese carbonate was noticed to create some heterogenous particles like MgO, Al_4C_3, and Al_8Mn_5 in melted form and agitation in melted form of carbon dioxide by decay of manganese carbonate and it was also noticed that manganese carbonate can be used as refiner of grains for Mg alloys and serves as super refiner for grains as compared to other grain refiners [37]. The inclusion of Mn in the Mg-Al alloy has lead us to know that it increases the volume fraction of the particles and reduces the size of the particles due to which we noticed good improvement in the PSN effect while material was carried through the procedure known as extrusion and the nucleation of the DRX was done by applying and the development of the particles after DRX was concealed by the huge ratio of the particles having nano size [38]. It was also seen that the yield strength was connected to the nano particles and UFG structure within the irregular directions and the existing mechanical properties were also noticed to be improved as compared to the that properties before addition of manganese [38].

2.3. THE EFFECT OF LA ON THE MICROSTRUCTURE AND MECHANICAL PROPERTIES OF MG-AL BINARY ALLOYS

Before the addition of La, the microstructure of as-cast material (Mg-4Al) was noticed to have only α particles along with the boundaries of the grains of huge β phase particles but after the addition of La to it, it came to be noticed that Al11La3 phase came into being and it made improvements in the sturdiness and resistance to wear of the Mg-4Al alloy and none of the phases like Mg17Al12 was noticed [39]. Mg-4Al alloy in as-cast condition was treated with La and gradual increment in the thermal conductivity of it was noticed because by the addition of La Al11La3 came into being which absorbed the solutes of the Al and extrusion process was also conducted and as a result, some more development in the thermal conductivity was noticed because handsome amount of solutes of Al got to be dissolved in solution of Mg during the process of extrusion and after the extrusion, the alloy was noticed to carry supreme properties such as thermal conductivity, more than 8% of elongation to failure, more than 250MPa of yield strength, more than 330MPa of ultimate tensile strength and super sturdiness [40].

2.4. THE EFFECT OF GD ON THE MICROSTRUCTURE AND MECHANICAL PROPERTIES OF MG-AL BINARY ALLOYS

By the addition of 0.66 wt.% of rare earth element known as Ga, it came to be revealed that it has capability of refining the α Mg from about more than 490 µm to less than 210 µm and can alternate the morphological properties of the phase known as Al11RE3 and the material was found to have increment in EL and UTS properties too [41]. The addition of Gd to an alloy known as AZ31 provides improvements in the alloy for hot rolling and provides reduction in the time at the time of homogenization [42].

PART 3

EXPERIMENTAL STUDIES

3.1. MATERIALS

In this study, AT31 Mg alloy and modified AT31 Mg are used as main materials. The raw rolls used for production are given in Table 3.1. Pure Mg, pure Al, pure Sn were obtained from Turkey, and master alloys were supplied from China. For production, a custom-made low-pressure permanent mold casting method was used (Figure 3.1) and the casting conditions given in Table 2. have been complied with. First into the stainless-steel crucible pure Mg was added. 1 hour was the waiting time when the temperature of 775 °C is reached. Afterwards, pure Al and then master alloys were added to the crucible. Meanwhile, the molten metal in the pot was stirred continuously. Final alloy addition to pure Sn crucible was added and after 10 minutes of mixing, the molten metal was 350 °C under 2-3 atm pressure. The stainless-steel metal with high temperature was injected into the molds.

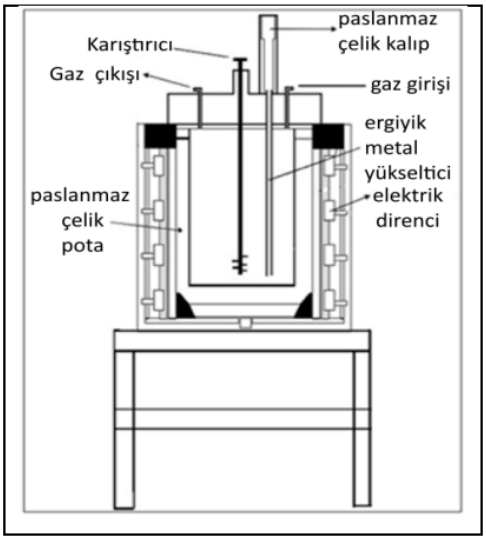

Figure 3.1. Diagram of low pressure permanent mold casting furnace.

Table 3.1. Casting conditions.

Shielding gas	Melting Temp: (°C) – Standby time (minutes)	Mold Temperature (°C)	Runner Temperature (°C)	Casting gas pressure (atm)
Argon	775-60	350	350	2-3

Table 3.2. Raw materials used for production (in % by weight).

Raw Material	Mg	Al	Sn	Mn	La	Gd
Pure Mg	%99,9	-	-	-	-	-
Pure Al	-	%99,9	-	-	-	-
Pure Sn	-	-	%99,9	-	-	-
Guage Mn	%90	-	-	%10	-	-
Guage La	%70	-	-	-	%30	-
Guage Gd	%75	-	-	-	-	%25

The chemical contents of the alloys produced are given in Table 6.3. For the chemical analysis process, Rigaku ZSX Primus II device belonging to Karabuk University Iron and Steel Institute XRF laboratory was used.

Table 3.3. Produced alloy groups (%).

Alloys Group	Mg	Al	Sn	Mn	La	Gd	Abbrevations
Mg-2.5Al-1.0Sn-0.3Mn-0.4La-0.16Gd	Bal.	2.53	1.04	0.37	0.36	0.16	A
Mg-2.5Al-1.0Sn-0.3Mn-0.4La-0.66Gd	Bal.	2.4	1.03	0.31	0.41	0.66	B
Mg-2.5Al-1.0Sn-0.3Mn-0.4La-1.33Gd	Bal.	2.4	0.99	0.35	0.39	1.33	C

3.2. HOT ROLLING

Billets with dimensions of 120x36x12 mm after casting were subjected to homogenization heat treatment at 400 °C for 24 hours. Materials were buried in sand to prevent metal oxidation and homogeneous temperature distribution during homogenization. Hot rolling process with parameters in Table 3.4 was applied to homogenized materials. Here, 20% (total 8 passes) section narrowing were applied for each pass. Inter-pass materials were kept in an oven at 400 °C for 5 minutes. Billets with an initial thickness of 12 mm total 83%.

After narrowing the section, it was reduced to 2 mm thickness. The AT31 alloy given in Table 3.4 has the rolling speeds of 1.5 m/min, 4.7 m/min and 10 m/min given in Table 3.1 for each of the La and the La-Gd groups.

Figure 3.2. Hot rolling machine used for rolling process.

Table 3.4. Rolling parameters.

Rolling temperature (°C)	Amount of section shrinkage per pass (%)	Rolling speed (m/min)	Total number of passes	Total section narrowing (%)	Initial and final thickness (mm)
400	20	1.5	8	~83	12-2
		4.7			
		10			

Table 3.5. Abbreviations for rolled specimen.

Specimen	1.5 m/min	4.7 m/min	10 m/min
A	A1	A2	A3
B	B1	B2	B3
C	C1	C2	C3

3.3. MICROSTRUCTURAL EXAMINATIONS

3.3.1. Sample Preparation

Acetic-picral (70ml ethanol, 5ml acetic acid, 5 gr picric acid and 10 ml distilled water) dispersant was prepared for the casting, homogenized and post-roll microstructure investigations of the produced alloys. Before the etching process, sanding with 600, 800, 1000, 1200, 2500 sandpaper and polishing with 1µm diamond suspension and 1µm felt was carried out.

3.3.2. Optical and Scanning Electron Microscope Images

Casting state homogenized and post-rolling microstructure investigations of the produced alloys Nikon Eclipse MA200 model optical microscope and SEM in Karabuk University (KBÜ) Iron and Steel Institute Metallography laboratory. It was carried out in the Carl Zeiss Ultra Plus Gemini Fesem model scanning electron microscope device in the laboratory. In addition, energy dispersion spectrometry (EDS) studies were carried out in the same SEM device.

Figure 3.3. Scanning electron microscope used for microstructure investigation.

3.3.3. X-ray Diffraction (XRD) and X-ray Fluorescence (XRF) Analysis

Rigaku branded ZSX Primus II model XRF and Ultima IV model XRD devices were used in the determination of the chemical contents and phases-texture of the produced alloys, respectively, in the XRD-XRF laboratory of KBU Iron and Steel Institute. XRD plots were taken on a copper-targeted XRD device in the range of 15-90°.

Figure 3.4. XRD machine for XRD investigation.

Figure 3.5. XRF machine for XRF investigation.

3.3.4. Particle Size and Twinning Fraction Analysis

Grain size analyzes were calculated from 200X magnification optical microscope images according to ASTM E112 standard. SEM images with 500X magnification were used to calculate the twinning fraction and point calculation was applied according to ASTM E562-02 standard.

3.4. MECHANICAL EXPERIMENTS

3.4.1. Hardness Tests

The hardness tests were carried out in BMS branded brinell hardness tester, based on the brinell hardness method, using a 2.5 mm diameter ball under 187.5 kg load, in accordance with the TS-EN-ISO 6506-1 standard, in the Micro and Macro Hardness measurement laboratory of the KBÜ Iron and Steel Institute. At least 3 measurements were taken from each material.

3.4.2. Tensile Tests

Tensile tests are carried out in the static laboratory of KBU Iron and Steel Institute, according to ASTM A370-12a standard, with a speed of 1 mm/sec at room temperature with MTS brand 100kN Servo Hydrolic. It was carried out using the Dynamic Tester. At least 3 tests were taken from each material.

PART 4

RESULTS AND DISCUSSIONS

4.1. MICROSTRUCTURAL EXAMINATIONS

In this section, the casting state, homogenization, and microstructure thinning (OM, SEM and XRD) of the produced materials are given.

4.1.1. Optical Microscope Images

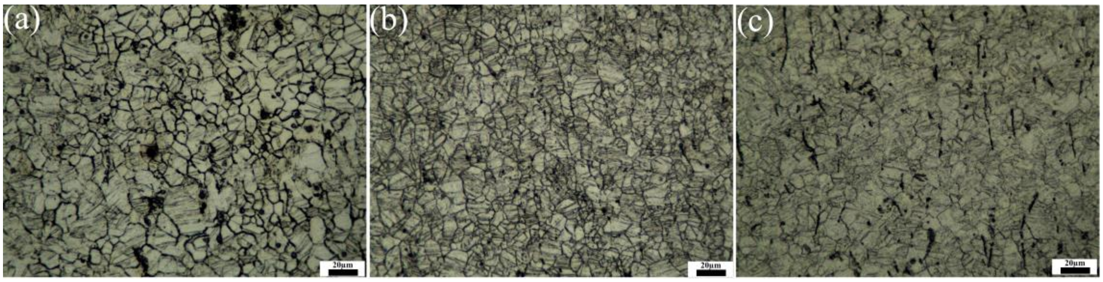

Figure 4.1. Optical microscope images of specimen (a) A1, (b) A2, and (c) A3.

In Figure 4.1. optical microscope images are given after rolling the AT31 alloy at 20% deformation rate and at rolling speeds of 1.5 m/min, 4.7 m/min and 10 m/min.

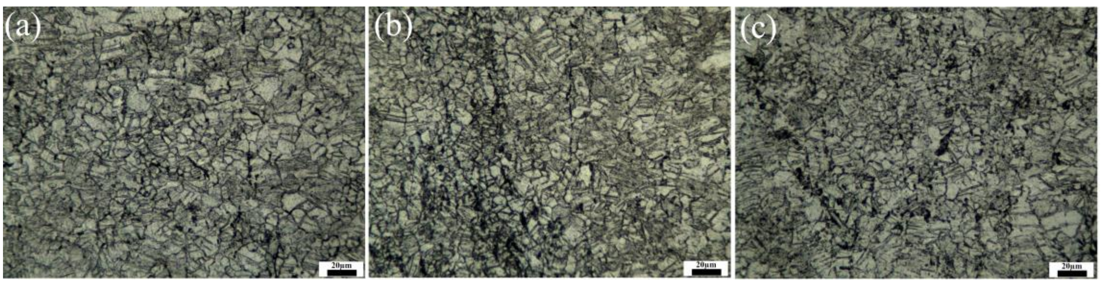

Figure 4.2. Optical microscope images of specimen (a) B1, (b) B2, and (c) B3.

Twinning occurred in the grains of AT31 Mg alloys, which underwent 20% deformation per pass at rolling speeds of 1.5 m/min, 4.7 m/min and 10 m/min. However, AT31 Mg alloy, which has undergone 20% deformation at a rolling speed of 4.7 m/min and 10 m/min, contains more recrystallized grains.

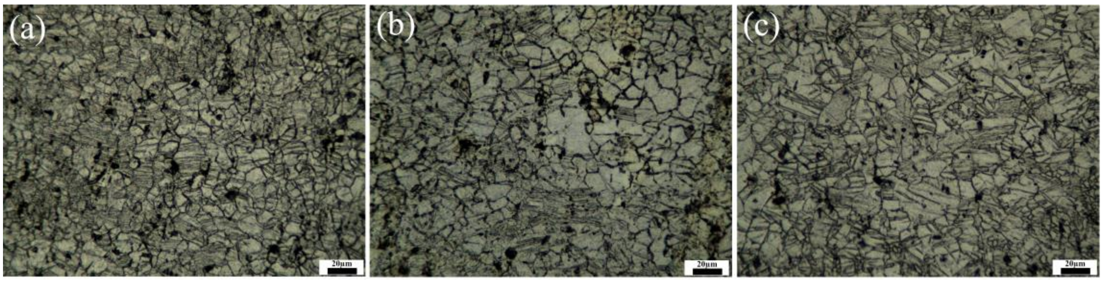

Figure 4.3. Optical microscope images of specimen (a) C1, (b) C2, and (c) C3.

Optical images of AT31 Mg alloy with 1.33% Gd addition, which underwent 20% deformation per pass at 1.5 m/min, 4.7 m/min and 10 m/min rolling speeds, show fine grains and twinning in some coarse grains. However, recrystallized grains can be detected from the optical microscope images. In addition, it is seen that materials deforming 20% per pass contain coarser grains with increasing rolling speed.

4.1.2. SEM Images

SEM images of rolled materials are given, respectively.

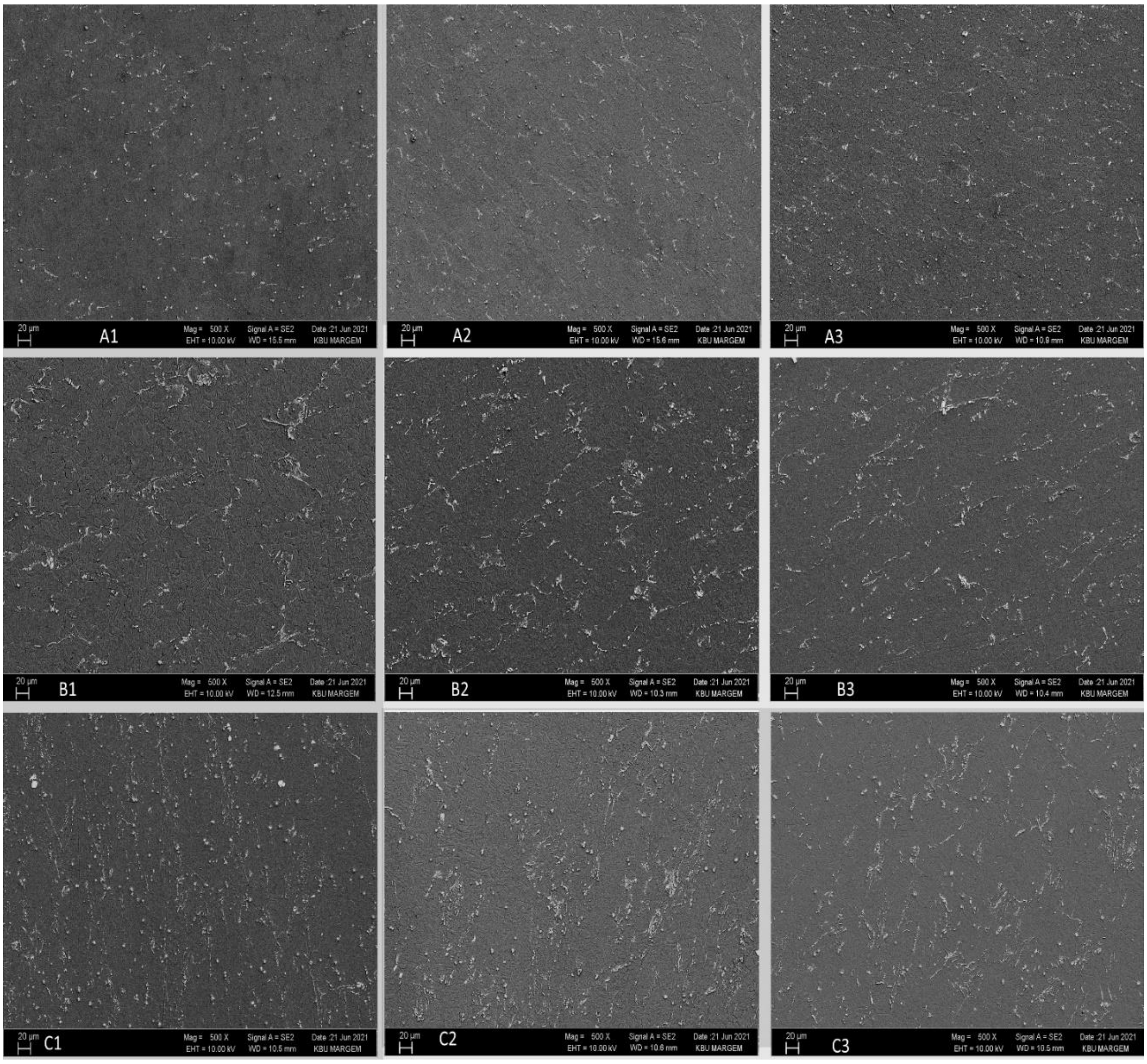

Figure 4.4. SEM images of rolled materials.

In Figure, SEM images are given after rolling the AT31 alloy at 20% deformation rates and at rolling speeds of 1.5 m/min, 4.7 m/min and 10 m/min.

As can be seen in Figures, widely dispersed polygonal and spherical secondary phases were formed in AT31 Mg alloys, which underwent 20% deformation per pass at a rolling speed of 1.5 m/min, 4.7 m/min and 10 m/min. In addition, there are larger spherical shaped secondary phases at a rolling speed of 10 m/min than at a rolling speed of 4.7 m/min. However, AT31 Mg alloys, which underwent 20% deformation at rolling speeds of 4.7 m/min and 10 m/min, widely dispersed thin spherical shaped secondary phases formed in a certain area, respectively. Here, it is seen that with increasing rolling speed, the secondary solutions line up along the grain boundaries and dissolve in the matrix.

4.2. TENSILE TEST

Tensile test results of AT31 with La and Gd rolled with 20% deformation rate per pass and rolling speed of 1.5 m/min, 4.7 m/min and 10 m/min are given in the table below.

Table 4.1. Tensile strength table.

SAMPLE	Dimension	Peak Load (kN)	Peak Stress (MPa)	Strain at Break (mm/mm)	Stress at Offset Yield (MPa)
A1	1.65x9.00	1.438	96.9	0.436	82.220
A2	1.72x16.95	7.249	248.7	1.861	158.319
A3	1.63x15.33	7.195	287.9	3.498	174.985
B1	1.80x15.50	2.630	94.3	0.492	90.413
B2	1.85x14.50	6.209	231.5	1.811	154.635
B3	1.82x13.30	6.102	252.1	3.812	155.079
C1	1.68x10.15	2.877	168.7	0.753	158.291
C2	1.65x8.40	3.454	249.2	1.511	176.743
C3	1.65x14.05	6.216	268.1	3.226	168.774

It can clearly be examined from the table that with increment in rolling speed, the peak load measured in kN, peak stress measured in MPa, strain at break measured in mm/mm, and stress at offset yield measured in MPa are also increasing but the value of C2 was greater than C3.

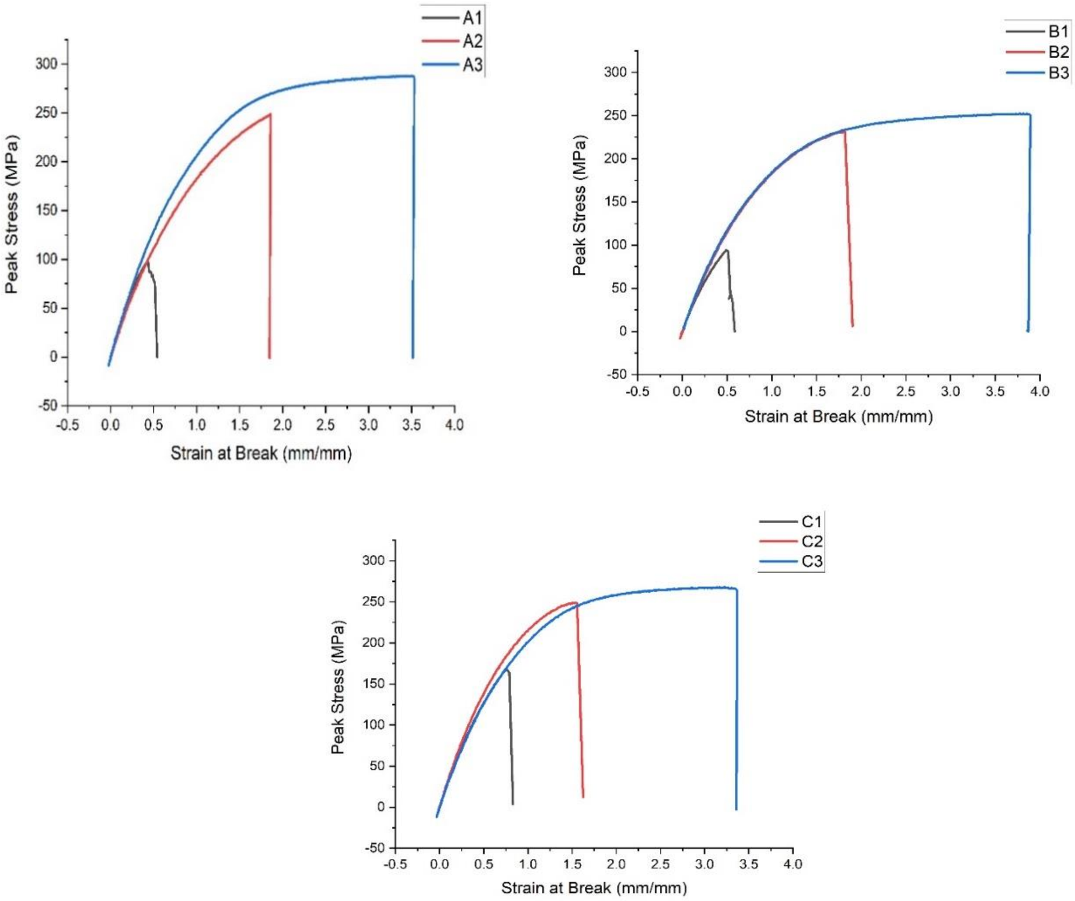

Figure 4.5. Tensile test graphs of specimen.

From the given graphs it can clearly be seen that the materials rolled at higher speed have maximum value of peak stress and strain at break. The black graph represents the materials rolled at a speed of 1.5 m/min, the red graph represents the materials rolled at a speed of 4.7 m/min, while the blue graph represents the materials rolled at the maximum speed of 10 m/min.

4.3. AVERAGE GRAIN SIZE ANALYSIS

Grain size analyzes of AT31 with La and Gd, rolled with 20% deformation per pass and at three different rolling speeds of 1.5 m/min, 4.7 m/min and 10 m/min, are given in the table and figure below.

Table 4.2. Average grain size table.

	AVG	STD	AVG	STD
A1	51.31056	9.2	25.65528	4.6
A2	34.15	1.5	17.075	0.75
A3	32.07	0.9	16.035	0.45
B1	39.07	3.07	19.535	1.535
B2	38.06	4.62	19.03	2.31
B3	35.01	9.11	17.505	4.555
C1	37.9	4.42	18.95	2.21
C2	34.9	3.13	17.45	1.565
C3	33.46	2.32	16.73	1.16

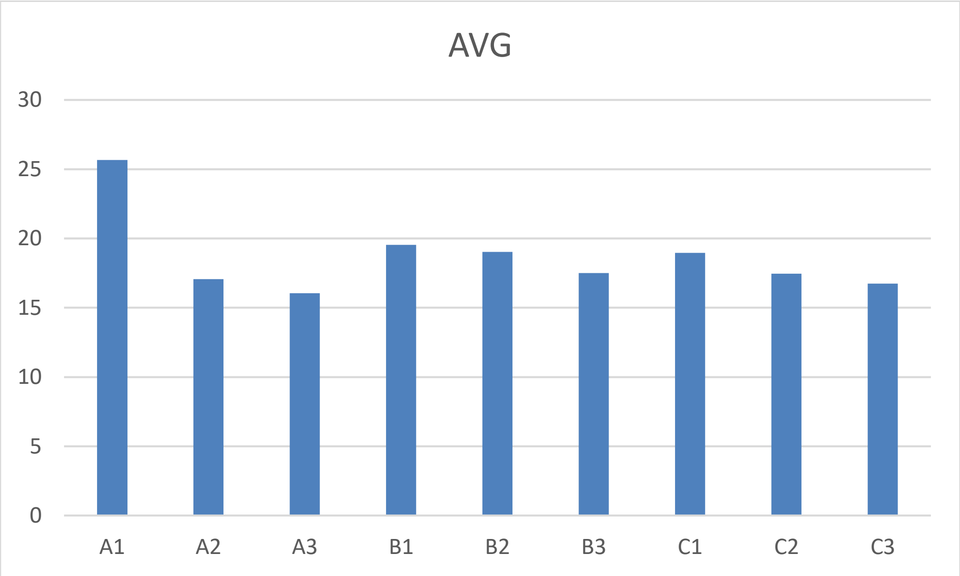

Figure 4.6. Average grain size graphs of rolled materıals.

The effect of rolling speed can clearly be seen in the table and figure that when we are increasing the speed of rolling, the average grain size of the materials is decreasing.

4.4. ULTIMATE TENSILE STRENGTH AND ELEONGATION

Ultimate tensile strength and elongation of AT31 rolled with 20% deformation per pass and at three different rolling speeds of 1.5 m/min, 4.7 m/min and 10 m/min, are given in the table and figures below.

Table 4.3. UTS and Elongation table.

	UTS	EL.
A1	82.22	0.436
A2	158.319	1.861
A3	174.985	3.498
B1	90.413	0.492
B2	154.635	1.811
B3	155.079	3.812
C1	158.291	0.753
C2	176.743	1.511
C3	168.774	3.226

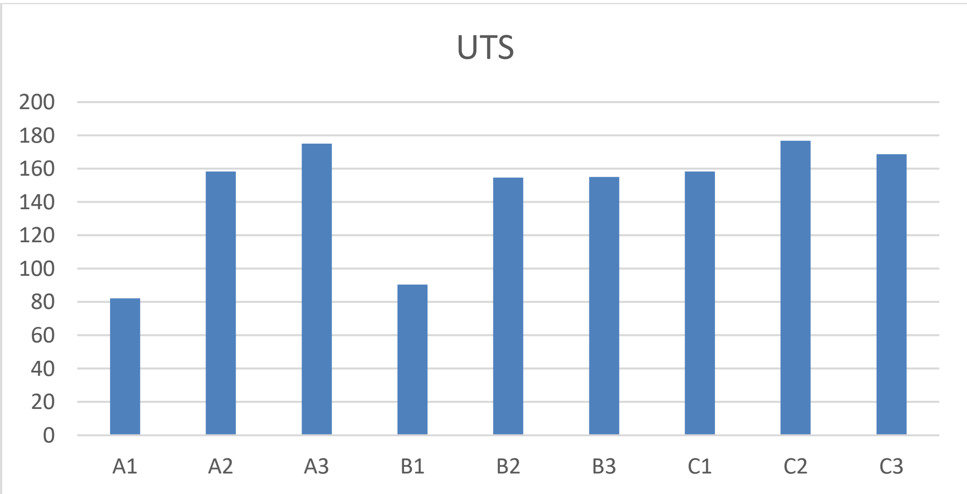

Figure 4.7. UTS graphs of therolled materıals.

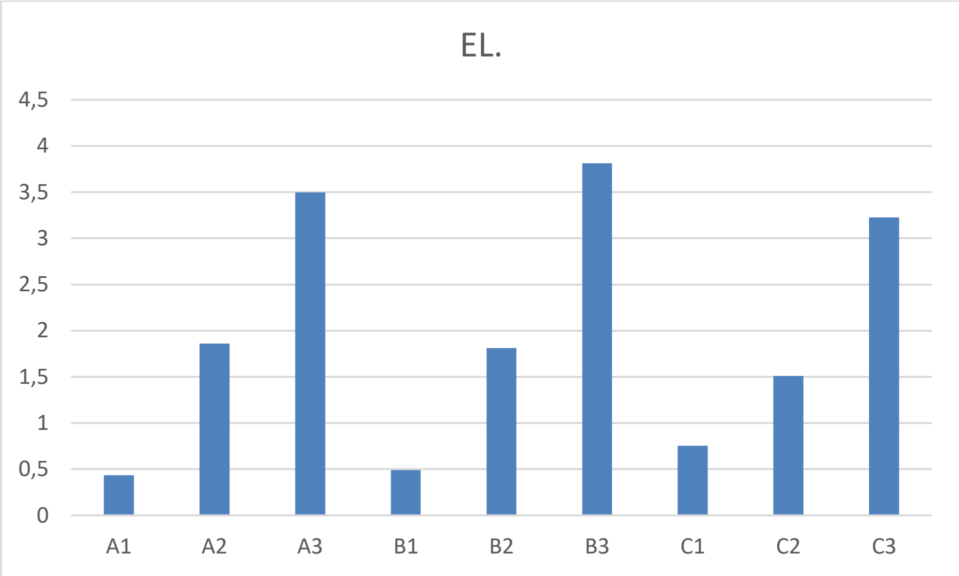

Figure 4.8. Elongation of the rolled materıals.

From the table 4.3 and figures 4.7 and 4.8 given above, we can examine that when the speed of rolling is increased the amount of ultimate tensile strength and elongation is also increasing while UTS of the specimens B2 and B3 remained same and the UTS of C2 was more than C3.

4.5. TWINS FRACTION

For the calculation of twins' fraction, (ASTM, E562-02) technique was taken in use. A 9x11 graph was drawn twice on the optical microscope image of each sample with a magnification of 50x. The average twins' fraction is given the table 4.4 below.

Table 4.4. Average twins' fraction.

				Average Twins' Fraction
A1	42	99		0.424242424
	42	99		
A2	27	99		0.196969697
	12	99		
A3	14	99		0.136363636
	13	99		
B1	17	99		0.151515152
	13	99		
B2	9	99		0.101010101
	11	99		
B3	8	99		0.090909091
	10	99		
C1	12	99		0.126262626
	13	99		
C2	9	99		0.095959596
	10	99		
C3	10	99		0.090909091
	8	99		

4.6. HARDNESS TEST

The hardness test results of AT31 with La and Gd rolled with 20% deformation per pass and rolled at 1.5 m/min, 4.7 m/min and 10 m/min rolling speed are given in Figure 4.9 and Table 4.5 below.

Table 4.5. Hardness test table.

A1	67.92333
A2	67.9
A3	59.41667
B1	63.33
B2	60.29333
B3	58.21
C1	65.25667
C2	64.28333
C3	58.26667

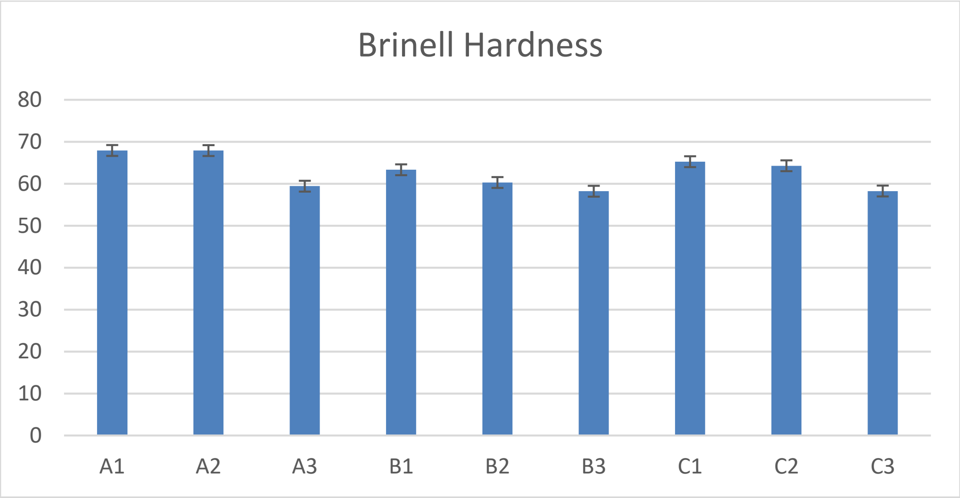

Figure 4.9. Brinell test graphs for the rolled materıals.

From the figure 4.9 and table 4.5, we can see that the Brinell hardness decreased with increasing rolling speed of AZ31 Mg alloy with 20% deformation per pass.

4.7. FRACTURE SURFACE ANALYSIS

The fracture surface analysis of AT31 with La and Gd rolled with 20% deformation per pass and rolled at 1.5 m/min, 4.7 m/min and 10 m/min rolling speed are given in Figures below.

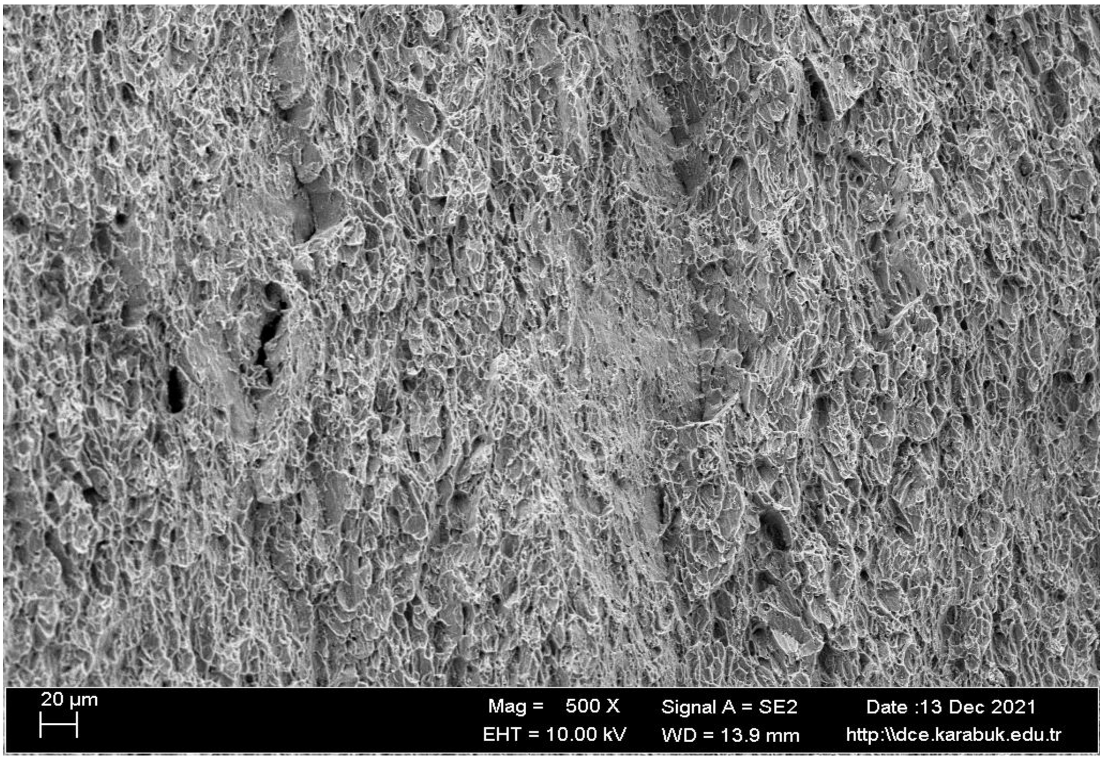

Figure 4.10. Fracture surface of A1.

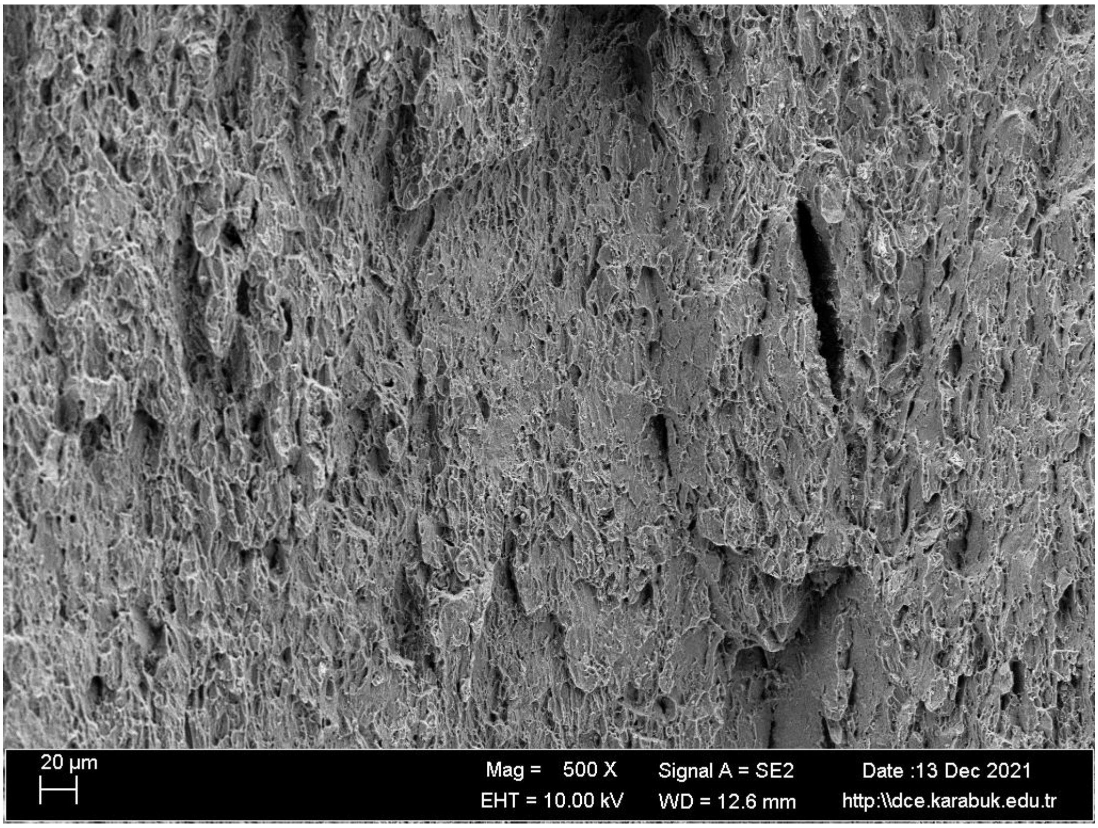

Figure 4.11. Fracture surface of A2.

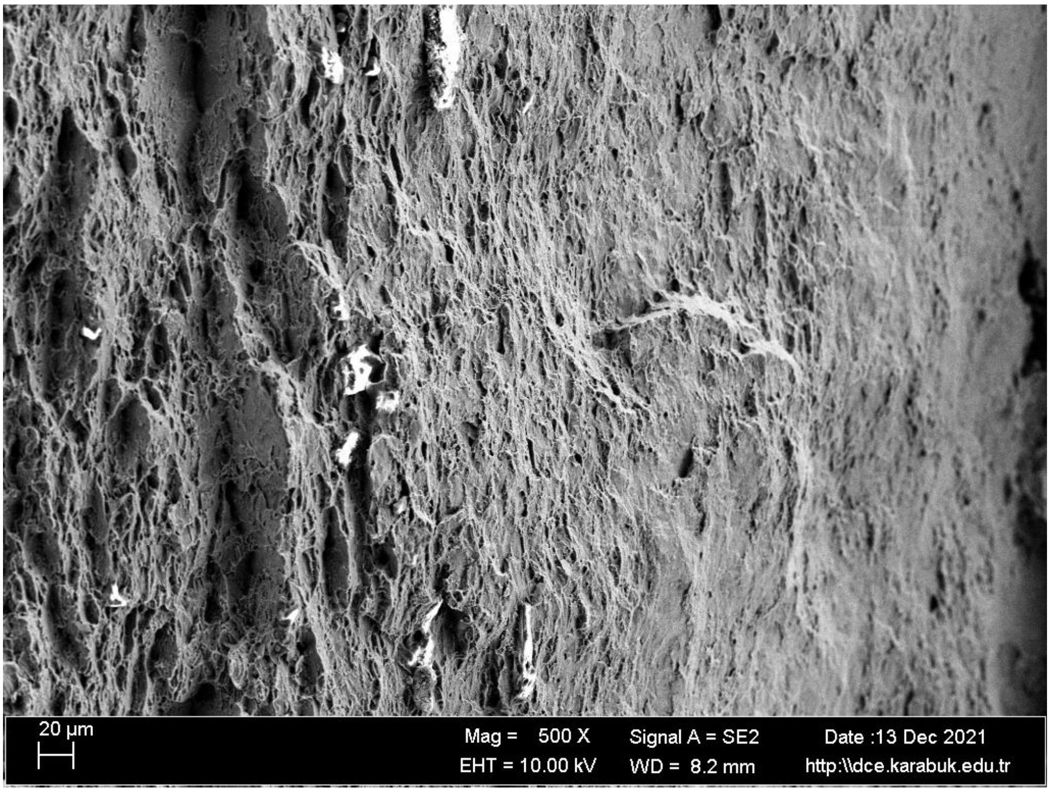

Figure 4.12. Fracture surface of A3.

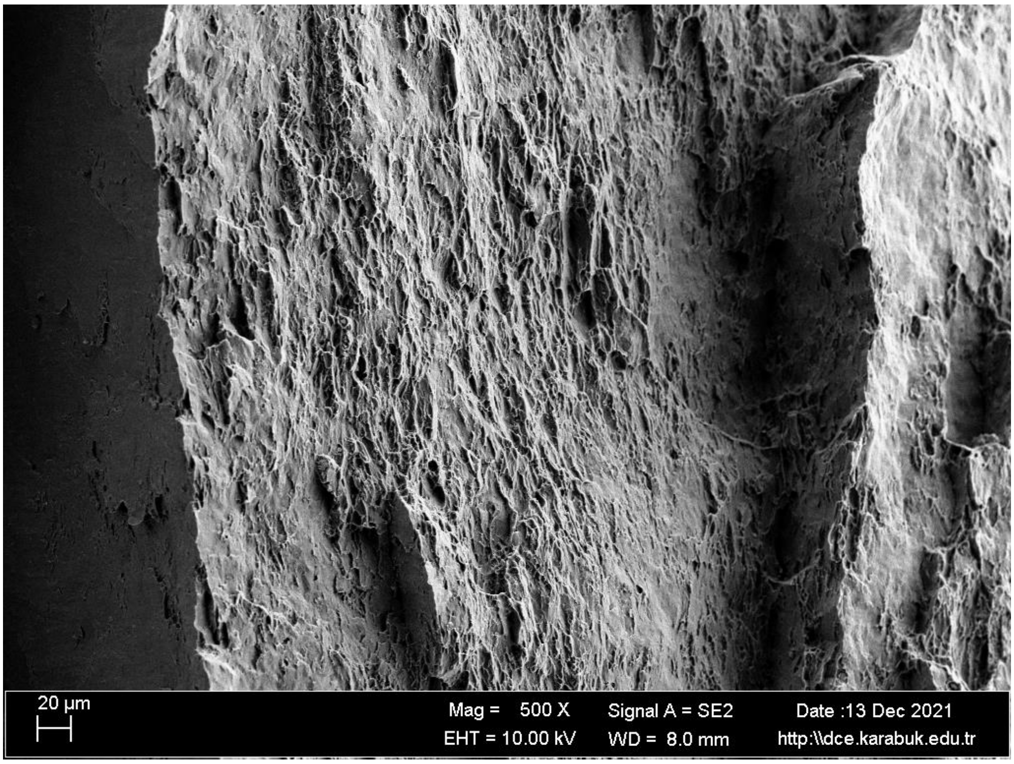

Figure 4.13. Fracture surface of B1.

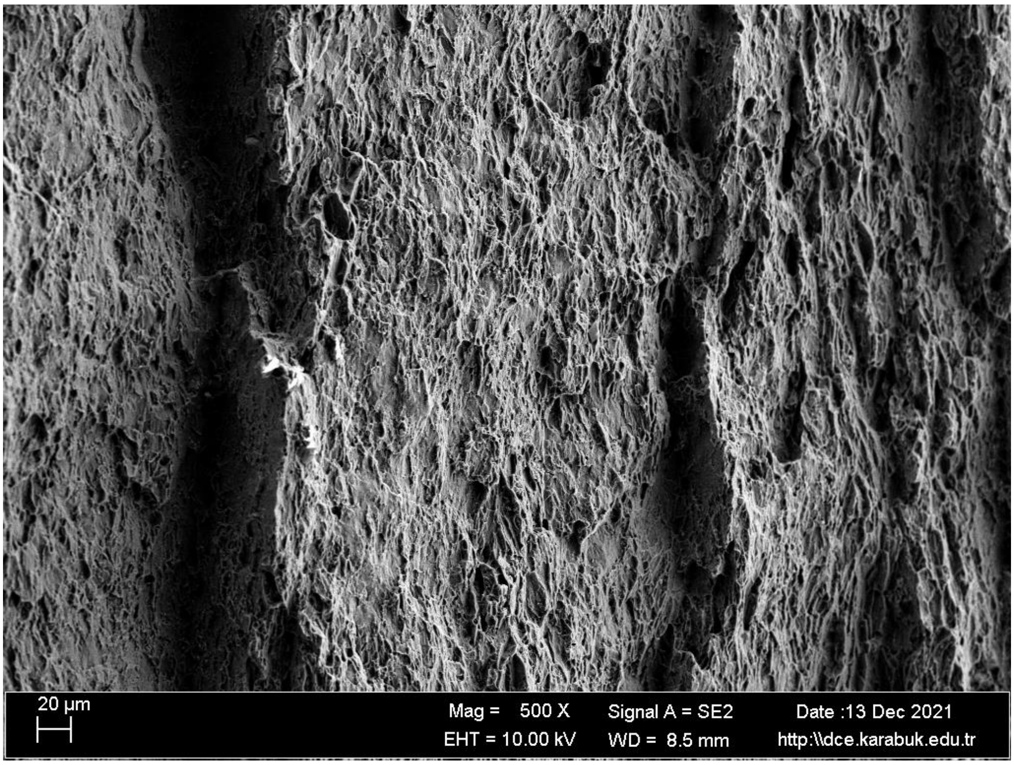

Figure 4.14. Fracture surface of B2.

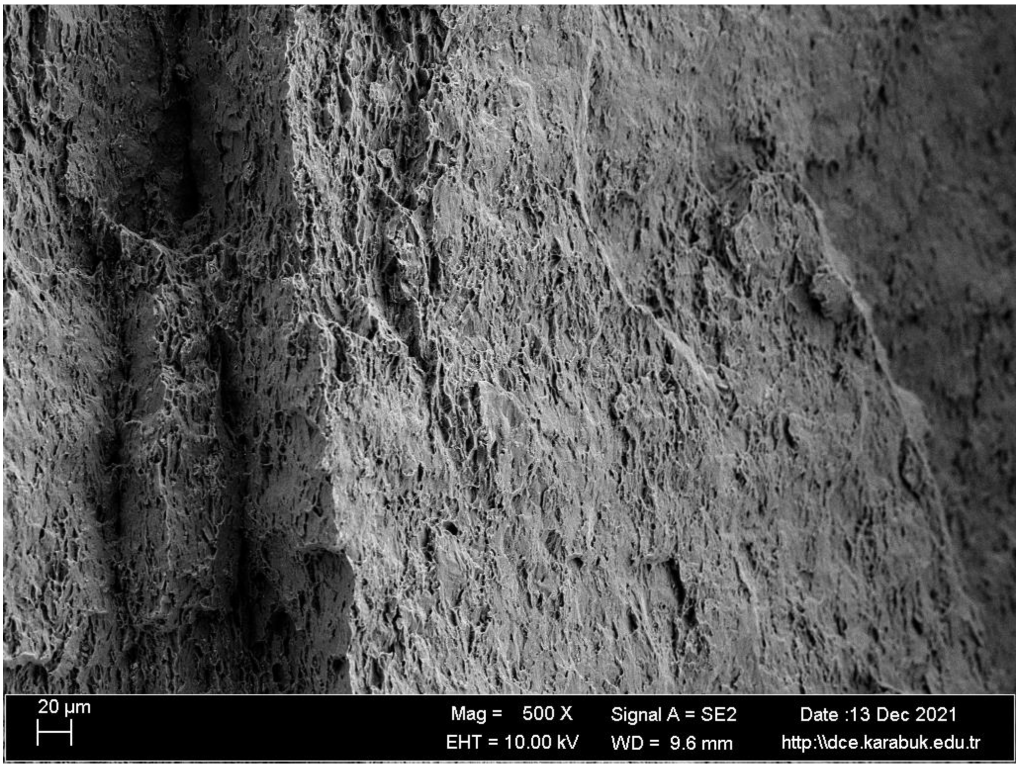

Figure 4.15. Fracture surface of B3.

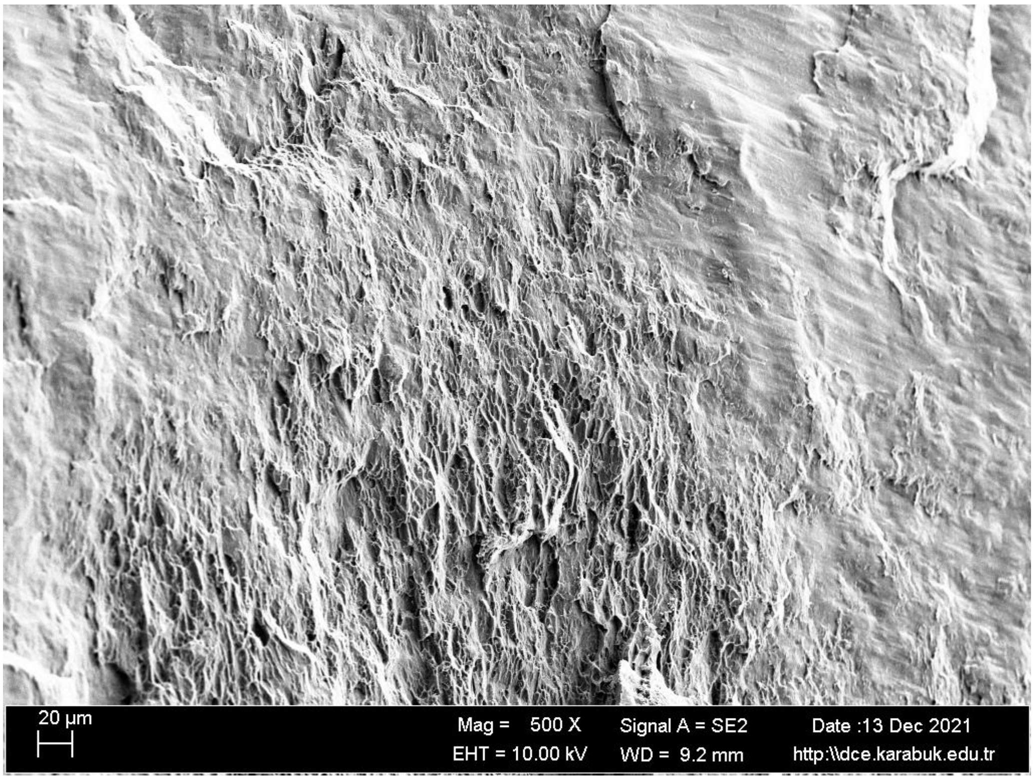

Figure 4.16. Fracture surface of C1.

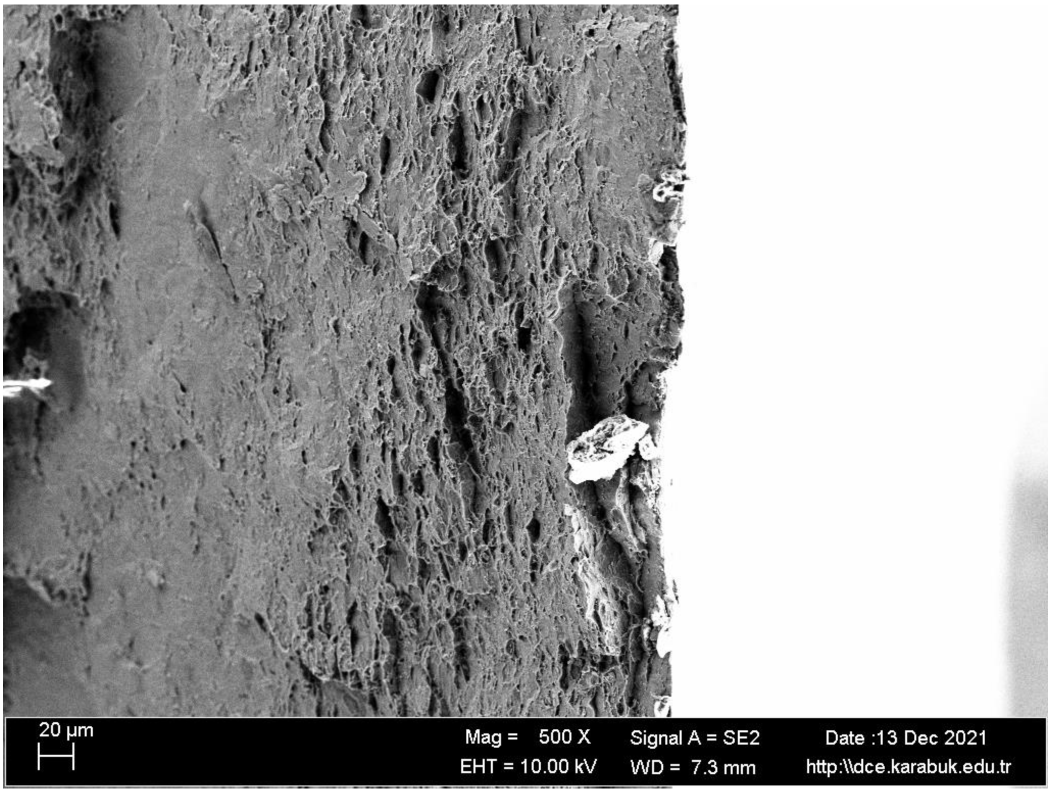

Figure 4.17. Fracture surface of C2.

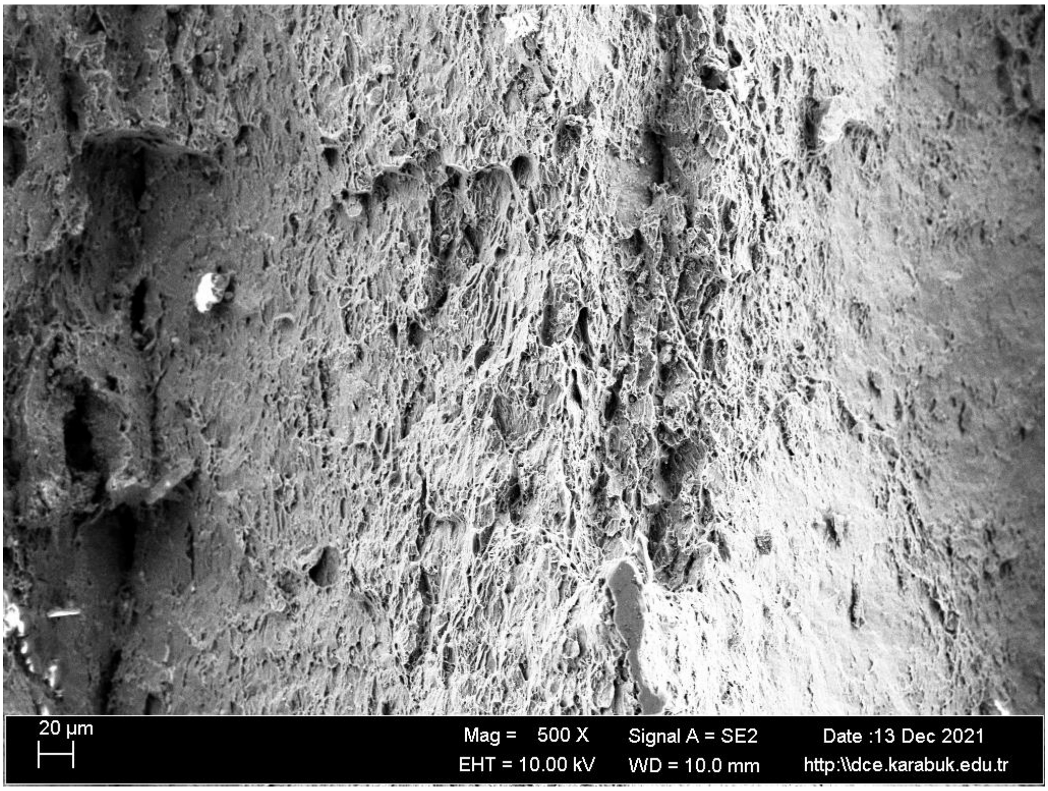

Figure 4.18. Fracture surface of C3.

The study about cleavage plane has been done by many people, for example, the cleavage plane occurred in Mg-9Al-1Zn alloy strip which was cold rolled [43]. From the images given above, we can examine that cleavage plane and dimples are occurring when material is passed through the hot rolling at different three speeds and we can clearly examine that the quantity of cleavage plane is more than that of dimples.

4.8. X-RAY DIFFRACTION (XRD)

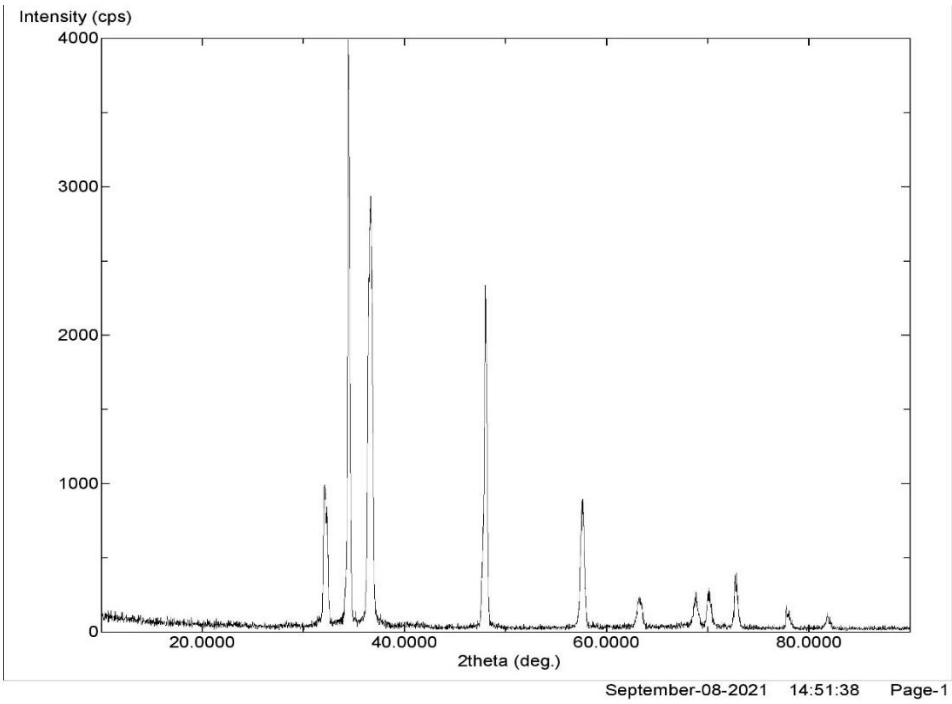

Figure 4.19. XRD pattern of specimen A.

The XRD results identified the intensity of Mg peaks, but the intensity of secondary phases was not identified by it.

4.9. CONCLUSIONS

Following conclusions can be drawn from this study:

- AT31 Mg alloy with 1.33% Gd addition, which underwent 20% deformation per pass at 1.5 m/min, 4.7 m/min and 10 m/min rolling speeds, show fine grains and twinning in some coarse grains.
- AT31 Mg alloys, which underwent 20% deformation at rolling speeds of 4.7 m/min and 10 m/min, widely dispersed thin spherical shaped secondary phases formed in a certain area, respectively.
- Average grain size, twin fraction and Brinell hardness decreased by increasing rolling speed.

- UTS was found to be increasing by increasing the rolling speed.
- Cleavage planes and dimples were found in the fracture analysis, but the cleavage plane was dominant to dimples.
- The intensity of Mg peaks was identified in XRD results.

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RESUME

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