

Master's Thesis

Investigation of Plastic instabilities in Aluminum Alloys



Degree program: Materials Science MSc

Student:

Charles Wambura Nyaketoleka

Supervisor:

Prof. Nguyen Quang Chinh

Eötvös Loránd University

FACULTY OF SCIENCE

BUDAPEST, 2021. January

TABLE OF CONTENT

1. Introduction 1	
1.1. Background	1
1.2. Problem analysis	2
1.3. Main objective.....	2
1.4. Specific objectives.....	2
2. Literature review.....	3
2.1. Strengthening and hardening mechanisms.....	3
2.2. Plastic instabilities.....	4
2.3. Solid solution homogenizing.....	6
2.4. Homogenizing temperature.....	9
3. Experimental materials and methods.....	11
3.1. Experiment plan for solid heat treating.....	11
3.2. Homogenizing of samples.....	11
3.3. Electrical polishing.....	13
3.4. Indentation tests.....	13
3.5. Acoustic emission.....	15
3.6. Pillar making process.....	17
3.7. Micro pillar compression test.....	18
3.8. True stress-true strain curve.....	20
3.9. Electron backscatter diffraction (EBSD).....	21
4. Results and discussion.....	23
4.1. Results for indentation tests.....	23
4.2. Results for micro pillars compression tests.....	31
5. Conclusion.....	40
6. Further studies.....	41
References.....	42

ACKNOWLEDGMENTS

I owe deep thanks to our great God for keeping us alive and our relatives and for his grace we completed this work. Moreover I would like to thank my Supervisor **Prof. Nguyen Quang Chinh** for his guidance and support in completing this work all the time, also he helped in doing indentation tests and analysis of all data after experiments, may God grant him all his wishes and long life

Furthermore I would like to thank all staff from both physics and chemistry departments for their support and guidance during my master studies and the knowledge I got from them which enabled me to complete this work, May God keep them alive and stay blessed.

Also I would like to thank **Mr. David Ugi** for his support in conducting experiments especially in Micro Pillars fabrication and compression tests that were very useful in this work, Stay blessed all the time. On other hand I would like to thank my father Mr. **Wambura Nyaketoleka** and his lovely wife **Ghati** for their full support from the beginning of my journey, May God grant them the long live.

Finally I would like to thank my beloved wife Miss. **Priccila Pilly** for her support and encouragement during the whole period of my studies, may God be with her all the time. Lastly but not least I would like to thank my friends and their wives from FFF group (**Mr. Makere, Mr. Mashango, Mr. Motatiro and Mr. Kiging**) for their cooperation and encouragement during my whole period of studies.

ABSTARCT

This work dealt with the study of plastic instabilities in Aluminium alloys based on mostly and widely used Al-Mg and Al-Zn-Mg-Zr alloys, which are the basic materials in the Al industry. The plastic instabilities in all alloys were carried out by indentation tests. Alloys were homogenized by heating them in electric Furnace at temperature of 500 °C for 30 minutes and quenching them in water to preserve the solid solution formed at the homogenizing temperature. In order to avoid the inducing of stresses or changing the properties of the sample, we used electrical polishing process to clean the sample before taking the indentation tests. The indentation tests were carried during storing the sample at room temperature (RT), and both the hardness of the samples and the character of the indentation curve were recorded and analyzed.

The experimental results have shown that the high purity Al-based Al-3Mg alloy is a stable solid solution. This sample always shows plastic instability, and its hardness is constant in all tests during storing at room temperature. The commercial purity Al- commercial purity showed the plastic instability but was not stable. The phenomenon of plastic instabilities taking place in this sample disappeared in the test carried out one day after quenching. Furthermore, its hardness was increasing during storing at RT. It should be noted that the commercial purity Al-3Mg alloy was harder than the high purity one. In the case of Al-Zn-Mg-Zr alloy, plastic instability was not observed at all due to formation of Guinier-Preston (GP) zones which formed rapidly in the material and suppressed the solute atoms. Because of the effect of GP zones, the hardness of this alloy is continuously increasing during storing at room temperature.

In the case of the stable solid solution high purity Al-3Mg alloy the phenomenon of plastic instabilities was investigated by micro pillar compression test, which was combined with the Acoustic Emission (AE) technique. This complex investigation was carried out by starting with micro pillars fabrication by focusing ions beam (FIB), then compressing the pillars by placing them in scanning electron microscopy (SEM) chamber. During the compression the elongation of pillars was measured by capacitive sensor, load, time, acoustic amplitude and acoustic energy were all recorded by computer. Both the global and local behaviors of the deformations of micro-pillars were analyzed and related to the AE signals.

1. INTRODUCTION

1.1. BACKGROUND

The unique combination properties of Aluminium and its Alloys make it to be widely used in World, due to its properties range from soft, highly ductility, light weight make the Alloys to be useful in structure metals. The density of Aluminium is about one third to that of steels. Apart from that also high strength of some Aluminium Alloys , oxidizing resistance, Corrosion resistance, good conductance , nontoxic add the more applications of that Alloys.

All Aluminium alloys respond to the thermal treatment based on phase Solubility, these treatments includes solution heat treatment, Quenching, Precipitation and Hardening. Few of them which are not good in the heat treatable are used in Cast or thermally modified Conditions unrelated to solution or precipitation effects.

Among of the Aluminium Alloys which are mostly known are as follows:

-Alloys in which Copper is the principle alloying element, used air craft due to its high strength

-Alloys in which Manganese is the principle alloying element, mostly used in architecture applications and various products

-Alloys in which Silicon is the principle alloying element, used in welding rods and brazing sheets

-Alloys in which Magnesium is the principle alloying element, mostly used in marine products

-Alloys in which Magnesium and Silicon are principle alloying elements, mostly used in automotive components

In this work I dealt with Aluminum Alloy in which the Principle element is Magnesium and Zinc. Magnesium is the major Alloying element in 5XXX series of alloys. Its maximum solid solubility in Aluminium is 17.4% but the one which is in application recently not exceed 5.5% Magnesium precipitates preferentially at grain boundaries as a highly anodic phase, which produces susceptibility to intergranular cracking and to stress corrosion. The addition of Magnesium increases the Aluminium in decreases in its ductility, high corrosion resistance and weldability.

In Aluminium-zinc-magnesium alloys there is the formation of GP zones and other metastable precipitate phases that change the microstructure as the precipitate becomes coherent to the Aluminium matrix. In this alloy the zone formation starts with the formation of Zinc or Zinc vacancies clusters. As Consequences of the opposite sizes effect of Zinc and Magnesium atoms in Aluminium, a strong interactive interaction might be effective even at a temperature of

solution treatment where in direct quenching the metal stable formation takes place by heterogeneous nucleation.

1.2. PROBLEM ANALYSIS

There is a tendency of Aluminium Alloys to show the plastic instability during the hardness tests, this research is aiming to investigate the Plastic instabilities in different Alloys during hardness tests after heat treating them and quenching them in water. The tendency of plastic instabilities in this Thesis was also investigated by Micro pillars test with AE technique

1.3. MAIN OBJECTIVE

To study and investigate the Plastic instabilities in Aluminium Alloys based on Aluminium-Magnesium Alloy high purity, Aluminium-Magnesium Alloy commercial purity and Aluminium-Zinc- Magnesium-zircon.

1.4. SPECIFIC OBJECTIVES

1. Determining the alloy type and its percentage in that Aluminium Alloy
2. Homogenizing of the Alloys
3. Quenching sample in water
4. Micro indentation tests
5. Checking the occurrence of plastic instability
6. Micro pillar compression tests by Acoustic emission technique
7. Conducting the Electron Back Scattered diffraction (EBSD)

2. LITERATURE REVIEW

2.1. STRENGTHENING MECHANISMS

In alloys the interstitial or substitutional impurities in solution cause lattice strain, as result these impurities interact with the dislocations strain field and hinder dislocations motion. Impurities tend to diffuse and segregate around the dislocation core to find atomic sites more suited to their radii, which reduce the overall strain energy and pin the dislocations.

The strengthening and hardening of Alloys can be achieved by different mechanisms as

a) Work hardening

This is the phenomenon of increasing hardness and strength of ductile materials as results of plastic deformation at a temperature far below its melting point. That plastic deformation at that condition leads to multiplications of dislocations which strain fields start to interact more closely, hindering the dislocations motion, dislocations generate stresses that trap other dislocations.

b) Grain size reduction

Owing to misalignment of the slip planes in adjacent grains a dislocation passing the grain boundary have to change its direction and thus loose its energy, a single grain which is in favorable orientation to slip cannot deform until the adjacent grains are also capable to slip. Therefore the small grains size the higher structural disorder and more slip barrier. This is well explained by Hall-Petch equation as $\sigma_y = \sigma_o + k_y d^{-0.5}$ where by σ_o is the minimum stress needed to induce dislocation glide in single crystal, k_y is the Hall-Petch slope and d is the diameter of grain. From the Hall-Petch equation the decrease in grain diameter (size) leads the increase in stress.

c) Precipitation hardening

In this process the hardening and strengthening of alloys is enhanced by the formation of extremely small uniform dispersed particles of second phase within the original phase matrix which accomplished by appropriate heat treatment in which the small particles of new phase participate lead a coherent precipitate forms that provides a substantial strengthening effect after the precipitate whose crystal structure and atomic arrangement continuous relationship with matrix from which the precipitate is formed.

The precipitation strengthening of supersaturated solid solutions involve the formation of finely dispersed precipitates during the aging heat treatment in precipitation process the saturated

solid first develops solute clusters which then become involved in the formation of transitional precipitates.

The mechanism of strengthening from precipitation involves the formation of coherent clusters of solute atoms, but the clusters still have the same crystal structure as solvent phase. The presence of precipitate particles and even more importantly the strain fields in the matrix surrounds the coherent particles, provides higher strength by obstructing the movement of dislocations.

The coherent or non-coherent precipitation phase is determined by closeness match or degree of disregistry between atomic spacing on the lattice of matrix and that of the precipitate. These changes in properties results from the formation of solute-rich microstructure domains or GP zones which is coherent to solvent matrix. The shape, size and distribution of GP zones depend on Alloy in which they form thermal and mechanical properties of the specimen.

2.2. PLASTIC INSTABILITIES

This is the process of repeated yielding during plastic deformation, it has been observed in metallic solid solution alloys also in precipitated strengthened alloys in under aged conditions. This phenomenon is observed by stress drop or steps beginning from the critical strain on original smooth stress-strain curves. The lower loading rate results to more rectangular steps than that of high loading rate (loading rate is the ratio of maximum force applied to critical time) The physical basis for appearance of PLC effect in negative strain rate sensitivity originated from the interaction between the mobile dislocations and diffusing solute atoms. The rate of work hardening becomes negatives for some points this implies sometimes the sample locally softer at increasing deformation.

The formation of GP zones after quenching, when the solution remains in solid for long time lead to the instabilities. In case of high solute content where the GP zones forms rapidly the disappearance of the plastic instability is connected to the effect of GP. The interaction of dislocations with GP zones become dominant compared to that of solute atoms therefore the formation of GP zones suppress the plastic instabilities, that lead the disappearance of plastic instabilities at certain state of decomposition of the alloys.

The interaction between mobile dislocations and diffusing solute atom, it can be expected that the solute concentration must exceed a certain minimum value of concentration. In Aluminium –magnesium alloys 0.45% is a minimum concentration of magnesium in order to get the instabilities. The instabilities occur when the equivalent strain rate is less than upper critical strain rate and the solute concentration is larger than critical concentration. In Aluminium –Zinc-Magnesium alloys are age harden able alloys. They form the super saturated solid solution; it

forms GP zones which are formed at room temperature that changes the microstructure and mechanical properties. The GP zones influence the strong occurrence of serrated yield point.

In case of higher solute content, the GP zones formed rapidly hence the interaction of dislocation with GP zones become dominant compared to that with solute atoms, therefore the formation of GP zones suppress the plastic instabilities. Consequently the plastic instabilities at a certain composition of the alloy.

In Aluminium–Zinc–Magnesium alloys the plastic instabilities is due to the interaction of diffusing solute atoms with the moving dislocations. The occurrence of the instability steps and corresponding micro hardness oscillations is in connection with solid solution phase of the alloys. The appearance and disappearance in micro hardness tests is due to decrease of solute concentration in solid solution matrix and varying the internal structure of the sample.

The disappearance is due to varying of microstructure, within the short time after quenching the formation of GP zones lead the alloy to be more a single phase solid solution. The GP zones formed in this case is not Strong enough to suppress the dynamic strain Aging effect. At this stage the GP zones effect on moving dislocations become dominant compared to that of solute atom hence cause the instability to disappear.

Most alloys show the instability behavior when the solute concentration exceed a certain amount for negative strain rate sensitive to appear, however up to date that critical or minimum concentration data is not available, even if the most indentation depth–load curves taken on Aluminium–Magnesium alloys for Magnesium concentrated 0.45wt% showed smoothly change no any instability observed. The same samples were also tested in two years later after quenching still have no effect on occurrence and development of steps on the indentation curves.

The disappearance of plastic instability can be due to either decrease of solute concentration in solid solution matrix or the varying in internal structure of sample. On other hand this disappearance can be caused by the formation of GP zone where the alloys become more in single phase solid that lead the GP zone to suppress the solute–dislocations interaction.

2.3. SOLID SOLUTION HOMOGENIZING

This refers to heating and cooling operations performed for the purpose of changing the mechanical properties and structures of these alloys. One of the essential attribute of heating treating alloys is temperature dependent equilibrium, solid solubility characterized by increasing solubility with temperature increase.

For having the precipitation hardening reaction we must first produce a solid solution that will enable the solid solution the maximum practical amounts of soluble hardening elements in the alloy. In this process the time at heating temperature should be long enough to form the

homogeneous solid solution. The heating temperature is limited by composition of alloys and the allowance is $\pm 6^{\circ}\text{C}$, but the heating temperature must range between the solvus and the eutectic melting temperature.

During the process overheating must be avoided because the Alloys that are overheated tends to change both in properties and metallurgical structures that the grain boundaries for overheated materials (above their melting points) are not detectable and the minimum temperature should be that temperature at which the complete solution occur. When the heating rate is higher non phenomenon of non-equilibrium melting must be considered and during the quenching before the liquid to equilibrate it will solidify and forms fine eutectic rosettes.

When the heating temperature is below the normal range solution is incomplete and that will the strength to be lowered than normal range due to reduction in concentration of the solid solution. The heating time should be long enough to enable the formation of homogeneous solution, this time depend on the size of the material or sample. During the heat treatment of Aluminium alloys the moisture contamination in the surface must be avoided because it will lead to the higher temperature oxidation that deteriorate the properties due hydrogen diffusion that affects surface layers during elevated temperature treatment, this is avoided by drying the sample or anodize the sample before heat treating it.

The solution heating process is followed by **Quenching** in order to develop the supersaturation.

Quenching is the process of soaking the alloys into liquid (mostly water or oil) so as to preserve the solid solution formed at the solution heating operation temperature by rapidly cooling to some lower temperature, usually near to room temperature, and maintaining the minimum number of vacant lattice sites to assist in promoting the low temperature diffusion required for zone formation.

The solute atoms that precipitate either on grain boundaries or other particles as well as vacancies that migrate to disordered regions. The solid solution is better to be quenched rapidly enough so as to produce supersaturated solution at room temperature. The volume of quenching media should be larger to avoid precipitation to occur during cooling.

Rapid quenching rate leads to the highest strength and good corrosion resistance and stress corrosion cracking; lower quenching rate leads to high ductility and fracture toughness. During quenching the residue stress also is developed due to thermal expansion during quenching, that is still warm central materials contract pulling in the already cooled part (outer part), the residue stress increases as the size increases, this problem can be avoided by tempering process after quenching or by quenching in a media that is warm or it is at a certain temperature.

According to the fact that in order materials to become harder it must be quenched so that to increase the cooling rate and to avoid the formation of pearlite that is soft. Rollason (1973) [6] gave out the types of quenching as follows:

Direct quenching

That is where hot material is immersed in a single cooling medium and cooled to the room temperature.

Two different media quenching

This where hot material is rapidly cooled in the third medium, usually water or brine to a temperature about 250°C - 400°C , and then quickly transferred to the second medium with less severity such as oil where it is held until completely cooled. This technique leads structure formed to be harder and crack less.

Stepped quenching

This is where hot material is first quenched in a medium having temperature from 150°C - 250°C , above the marten site transformation point. The work piece is held until it reaches the temperature of medium; usually the molten salt bath throughout its whole cross section and finally the work piece is transferred to an oil bath and cooled to the room temperature.

The most preferred types of quenching are:

Two different media quenching

This method is preferred due to the following reasons

- i. It relieves internal stresses of the work piece
- ii. It less distortion
- iii. It is easy to conduct
- iv. It has minimal errors during performance

Direct quenching

This method is selected because:

- i. It is easy to conduct
- ii. Less danger for hardening cracks being formed
- iii. It less distortion

Structure of quenched Alloy is affected by the cooling rate; the cooling rates of various media are different. The quenching power of any medium is determined by:

- a) **Ability to conduct heat:** The media that has high conductance has high quenching capability due to the faster heat absorbing from the hot work piece.
- b) **Viscosity:** The media that has high viscosity has low ability to form vapor hence high quenching rate at the third and second cooling rate.

- c) **Specific heat:** The media that has high specific heat capacity has high quantity of heat therefore has high capability of absorbing heat from the hot specimen hence has high quenching capability.
- d) **Gasifying temperature:** The media that has the low gasifying temperature has high cooling rate because heat is carried away by the vapor rapidly due to low boiling point of that media.

The most used media are oil and water because they have high cooling rate, they cause less distortion and cracks and they facilitate stress relieve

According to Burton (1956) [3] regardless of the quenching medium and provided that the quenching rate is rapid, the center of the larger piece will cool appreciably more slowly than its surface.

This may causes the serious cracking in the heat treated part because the precipitation has greater volume than that from which is formed. After the surface has become hard and brittle, the center of the piece transforms and expands, often cracking the surface layers of material, this is called *quench cracking* and is quite prevalent when the quenching rate is high.

For two direct quenching techniques is better to start with water then followed by oil because at the third stage because water has high cooling rate that lead the formation of cracks while oil has low cooling rate at this stage that lead the formation of precipitation without cracks.

2.4. HOMOGENIZING TEMPERATURE

Homogenizing temperature is the temperature at which the solid sample is heated to form solid –solution in homogeneous form. The homogenizing temperature is determined from the phase diagrams, which vary according to composition; the temperature must be between the solvus and melting point for prohibition of both over and under heating during the solid solution heating process. Some examples of phase diagrams for different Al alloys can be seen below:

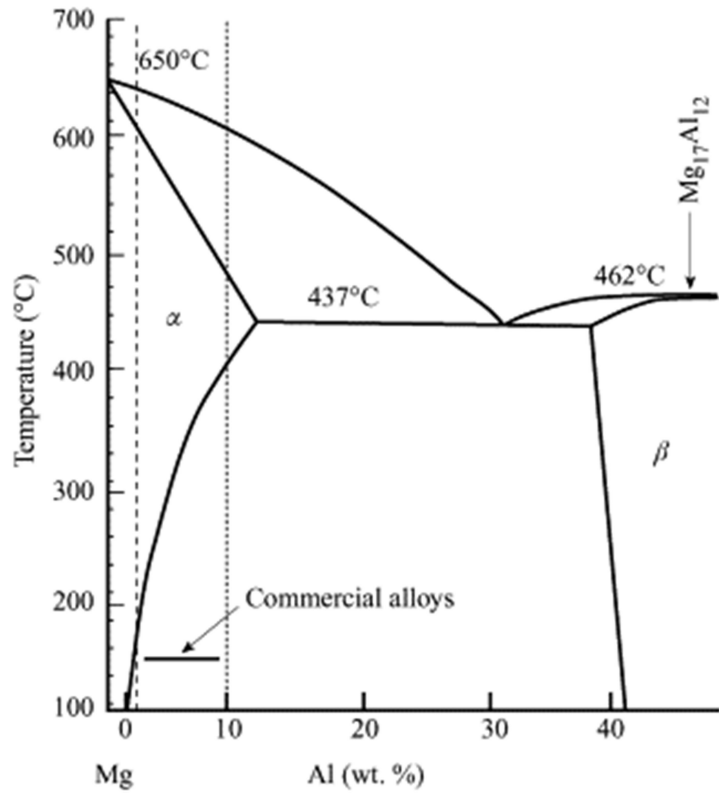


Figure 1: Phase-diagram of Aluminium magnesium alloys

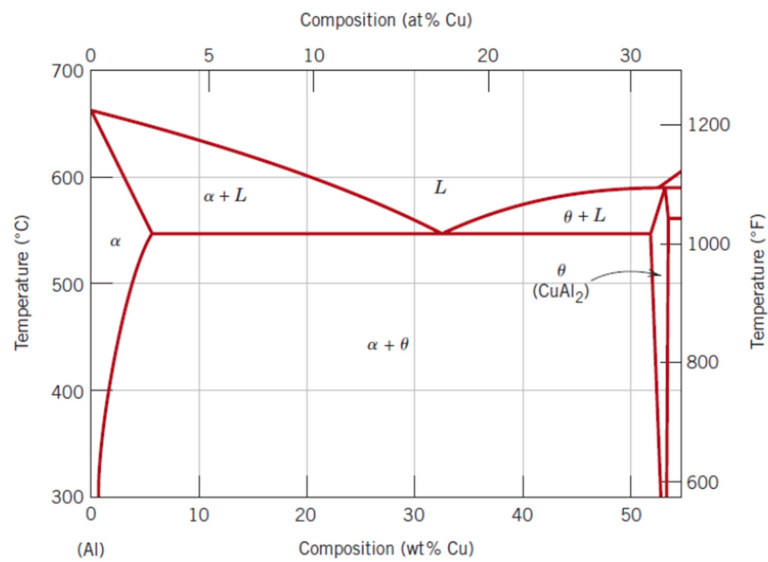


Figure 2: Phase-diagram of Aluminium copper alloys

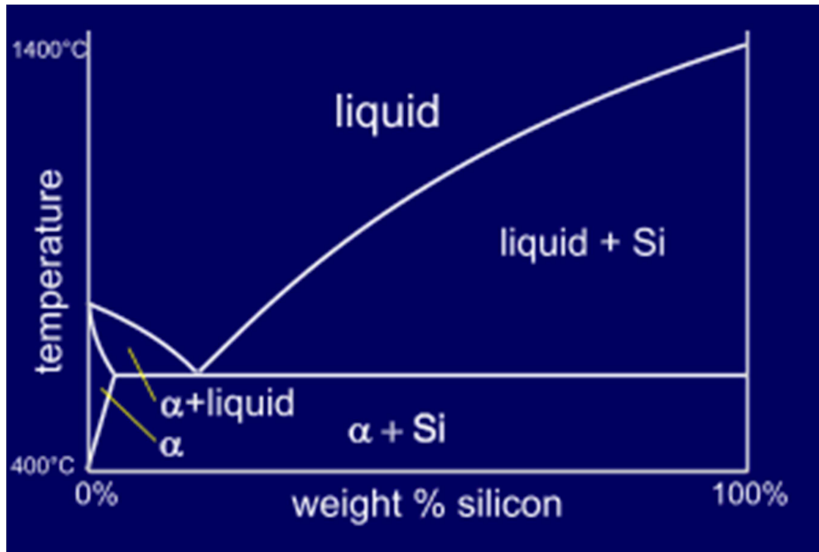


Figure 3: *Phase-diagram of Aluminium silicon alloy*

3. EXPERIMENTAL MATERIALS AND METHODS

3.1. EXPERIMENT PLAN FOR SOLID HEAT TREATING

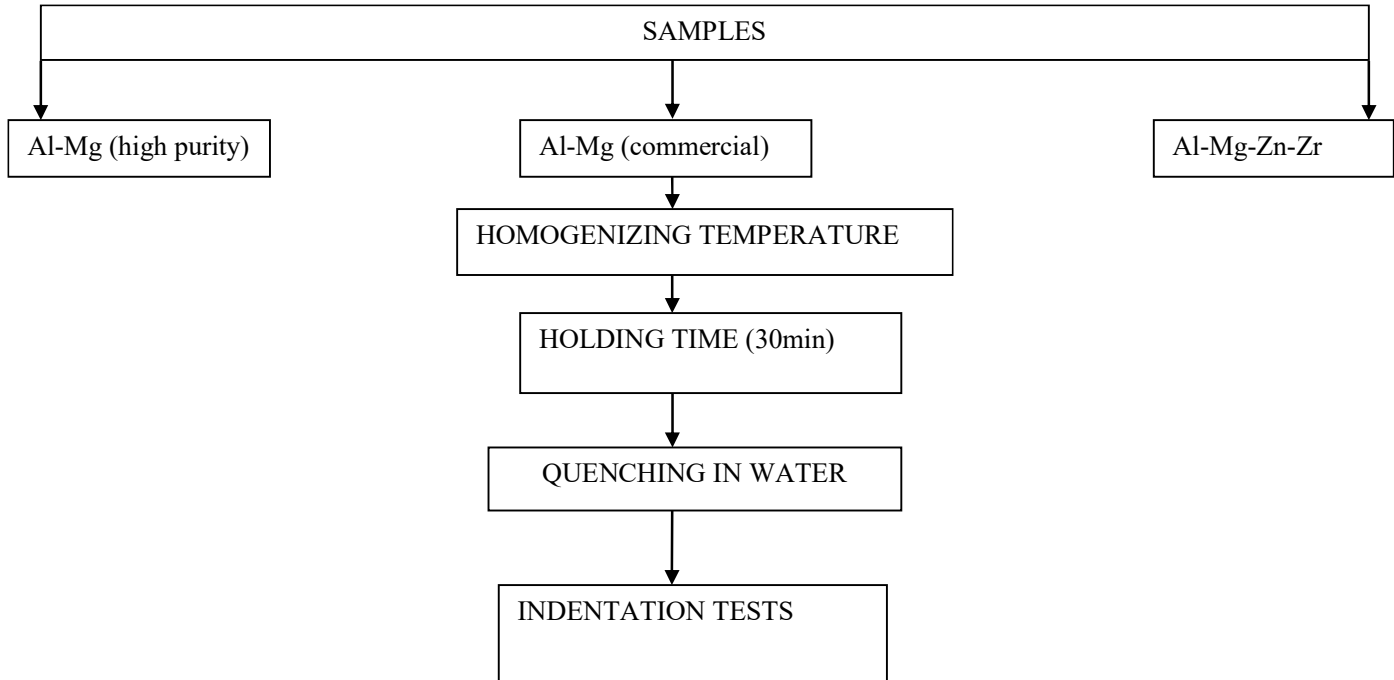


Figure 4: *Experiment Process for Indentation tests.*

The same procedures were repeated for all three samples.

Homogenizing temperature was determined from the curves in figures 1 and 2 depending on the composition by weight percentage and the type of Alloy. Indentation tests were carried in three interval of time for each sample, immediately after quenching, two hours after quenching and one day after quenching. Same procedures were applied for all three samples

In this study we dealt with three samples as shown in Experiment plan above:

- Aluminium-Magnesium Alloy high purity –Al-3Mg (in weight percent)
- Aluminium-Magnesium Alloy commercial purity – Al-3Mg
- Aluminium-Zinc- Magnesium-zircon – Al-4.8Mg-1.2Zn-0.14Zr

3.2. HOMOGENIZING OF SAMPLES

This involves the heating and quenching of samples at specified temperature

Heating-Soaking the sample at temperature sufficient high for specified time enough to achieve the nearly homogeneous solid solution. In our experiment the sample was heated in electric Furnace at temperature of about 500^oC for 30minutes



Figure 5: *Heating sample at 500^oC in Electric Furnace for 30minutes*

Quenching-Refers to soaking a sample into liquid so as to preserve the solid solution formed during heating process by rapidly cooling it to lower temperature. In our experiment the sample was cooled in water at Room temperature. The process was rapidly because the water container was placed under the Furnace, when the time required reached the lid at the bottom of the furnace was opened and the samples dropped into water container immediately.



Figure 6: *Quenching the Sample in water at Room temperature*

3.3. ELECTRICAL POLISHING

This is the electrochemical process that removes materials from metal workpiece, reducing the surface roughness by levelling micro peak and valleys and improving surface finishing.

In this process the work piece is immersed in temperature controlled bath of electrolyte and serves as an anode connected to the positive terminal of DC power supply. The negative terminal is being attached to the cathode. As current passes from the Anode where metal on the surface is oxidized and dissolved in electrolyte to the Cathode.

At the cathode the reduction reaction occurs that normally produces hydrogen, electrolyte used for electro polishing are concentrated acids having higher viscosity such as mixture of sulfuric acid with Phosphoric acid.



Figure 7: *Electro polishing the sample*

3.4. INDENTATION TESTS

This is the mechanical test that is useful in determining hardness of material or surface of materials that is applicable in design, structure or materials development. This is used to tell if the materials hardness is suitable for a certain application or to check the improvement of that material after treatment.

Hardness tells the ability of materials to resist permanent deformation like indentation, wear, abrasion and scratch. Micro hardness is when applied load is less than 1kg when the load applied is more than 1kg the last is termed as Macro hardness. For materials which are very thin

and small in size we use Micro hardness test. In this Thesis we will use the micro hardness method for testing in which the highly precision of instrument is needed. In Vickers Diamond Pyramid hardness number the applied load divided by the surface area of the indentation

This method is applicable for determining the mechanical properties of small volume materials without the complication of sample preparation. In this method hardness is determined perpendicular to the sample surface.

It studies the hardness changes during deformation under dynamic loading; due to this the hardness can be affected by loading rate. During the process an indenter penetrates into the surface of materials with the constant loading rate. When the maximum load is reached the load decreases with the same rate as for loading and the indenter moves backward. The load penetration depth curve is recorded in both loading and unloading part of the measurements. The characteristics features of these curves are used in determination of mechanical properties of solids.

The loading and unloading shapes from curves depend on geometry of indenter, loading rate, orientation of PLC (Portevin-Le Chatelier), testing temperature and mechanical properties of the material.

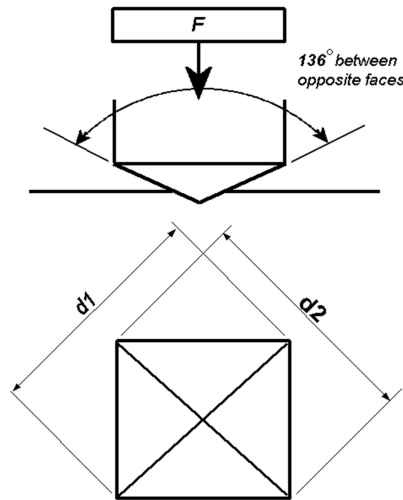


Figure 8: Geometry of Vickers indentation standard

The force applied relates to penetrating depth in a quadratic function

$$HV = 1.854 \frac{F}{d^2} \quad (1)$$

Where by F is the load applied

d is the arithmetic mean of two diagonals

In Knop hardness numbers the formula used is

$$KHN = \frac{F}{CL^2} \quad (2)$$

Where by

F is the load applied

L is the length of long diagonal

C is the constant (C=0.07028)

In the dynamic micro hardness test the indentation depth (h) and the load (F) are recorded as function of time by computer. Increasing the load at constant load rate ($F=kh^2$) where by the

$$HV = 3.8584 \left(\frac{\delta \sqrt{F}}{\partial h} \right)^2 \quad (3)$$

In our experiment of indentation hardness test samples were tested immediately after quenching, two hours after quenching and one day after quenching. All reading were recorded by the computer

In this test the loads and depths were recorded during loading and unloading process, loading process was recorded from the starting until we reach the maximum load.

For our experiment the maximum load was 500mN/s at loading rate of 10mN/s

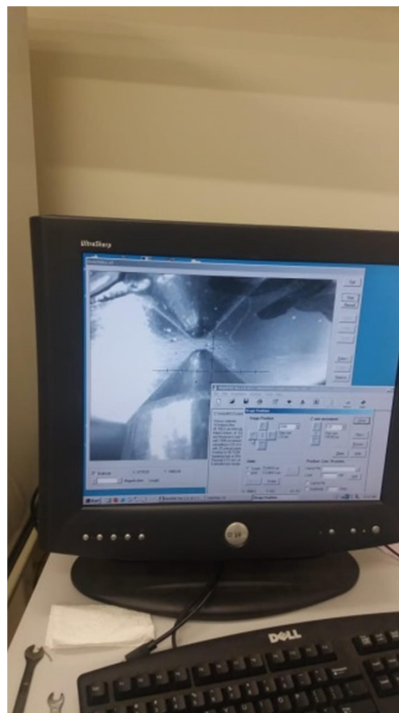


Figure 9: *Indentation testing*

3.5. ACOUSTIC EMISSION

This is the elastic energy spontaneously released during dynamic and irreversible changes of microstructure of materials. The Acoustic emission (AE) can be observed during the plastic deformation and phase transformations in materials. During the plastic deformation process there is dislocation motion that yields information on the dynamic process involved in plastic deformation of metals and alloys

The technique depends on the elastic energy released due to some irreversible changes of the microstructure. The AE source the released energy forms stress pulse which propagates through the material bulk as transient elastic waves then the stress is transformed into specific wave mode. These waves re detected by piezoelectric transducer which are coupled to the specimen surface either mechanical or through wave guide. This wave form of AE signal is significantly affected by source mechanism, propagation through the specimen volume and detection by transducer.

In continuous signal the amplitude does not fall under a certain threshold level during relative long period of time. The continuous signal is connected with micro mechanism of plastic deformation like dislocations glide, recrystallization, and phase transitions. The number of signals also affected with machine noisy. The AE is useful in monitoring of collective dislocations motion as well as deformation twinning because both are associated with dislocations dynamics.

During the process of dislocations movement some of energy is transformed to heat energy resulted from different interactions with photons and another part is lost without being detected. The fast operating dislocation source and sudden release of dislocations pile ups are in the background of detectable AE. The AE event rate is proportional to the change in density of mobile dislocations, they proposed an avalanche like dislocation break way model in which the dislocations are pinned at obstacles. The AE is generated when deformation stress reaches the value necessary for activation of first dislocation source subsequently the dislocations nucleate faster and move toward the next obstacles.

The following dislocations mechanism generate the AE

- a) Relaxation of stress field caused by passage of dislocations in which the transient part Of stress field of moving dislocation causes AE
- b) Annihilation of dislocations –the disappearance of stress field of annihilated dislocation causes AE
- c) Acoustic radiation of either accelerated or decelerated dislocations

3.6. PILLAR MAKING PROCESS

In micro compression tests we use pillars in micro scale which are fabricated by Focus Ions Beam (FIB) milling which enables the continuously visual control during the fabrication process. This process is time consuming and not useful for all materials due to the difficulty in controlling the dislocations density or weak forces connecting between the substrate and grown micro pillar.

There are two types of FIB milling

- FIB lathe milling: in this process the ion beam is almost perpendicular to the axis of micro pillar. The substrate is rotated during milling process and resulting in cylindrical micro pillar.
- FIB annular Milling: in this process the axis is parallel to the ion beam and the ion beam etches the surface of the substrate. In this is easy to control the final geometry of the micro pillar even though the ions implantation on the sample during processing can causes effects on mechanical properties of the sample.

Through this technique we are able to determine the fundamental plastic deformation of micrometer scale.

The sample for this test was prepared by FIB etching process as

- a) The sample was etched by orienting it so that the normal vector of the surface was parallel to beam direction at 30nA ions current.
- b) The thin layer of Platinum was deposited so as to ensure the ion beam to fabricate the smooth side surface of the pillar
- c) The stage was tilted by 7° in combination with 52° ion-electron beam angles which resulted 45° ion beam direction with respect to the surface normal with 5nA applied current.
- d) The steps repeated for all sides of the pillar so as to get the square pillar
- e) After completing one pillar all steps were repeated to other eight micro pillars



Figure 10: *Ones of the fabricated micro pillars (each pillar is 15 μ m long and square side of 5.25 μ m)*

3.7. MICRO PILLAR COMPRESSION TEST

In this test the intermittent stress-strain response originated in the repeated pile-up and break outs (pinning and unpinning) of dislocations from the solute atoms where these atoms act as obstacles for dislocations movements. Due to obstacles to mobile of dislocations the strong Acoustic signal with greater energies and amplitudes are generated.

Compression testing process

The testing device was placed inside the SEM chamber in which two linear ultrasonic motors positioned the sample in X and Y direction

The AE transducer was mounted on the top of this double stage, in Z direction two stages were used, linear step –motor stage for ran movement of the compressing tip closer to the sample. The Piezoelectric positioning (PEP) was mounted on the linear step motor stage, during the compression test only this stage moved. The standard spring was mounted on the PEP stage with higher transversal but very low longitudinal stiffness which used to measure the external force. The spring elongation was measured by capacitive sensor with 0.1nm resolution. The force, elongation, AE signals all were recorded by computer program.

The SEM was equipped with environmental stage so as to reduce the heating effect during test by controlling the sample temperature. To suppress the disturbances due to vibrations effects the strong permanent magnets were placed near lamellar of force measuring spring

During the compression test nine micro pillars were compressed by varying two parameters

- Loading rate , in which our loading rates were $0.003\mu\text{m/s}$, $0.01\mu\text{m/s}$ and $0.05\mu\text{m/s}$
- Full deformed micro pillars and that of only 20% deformation

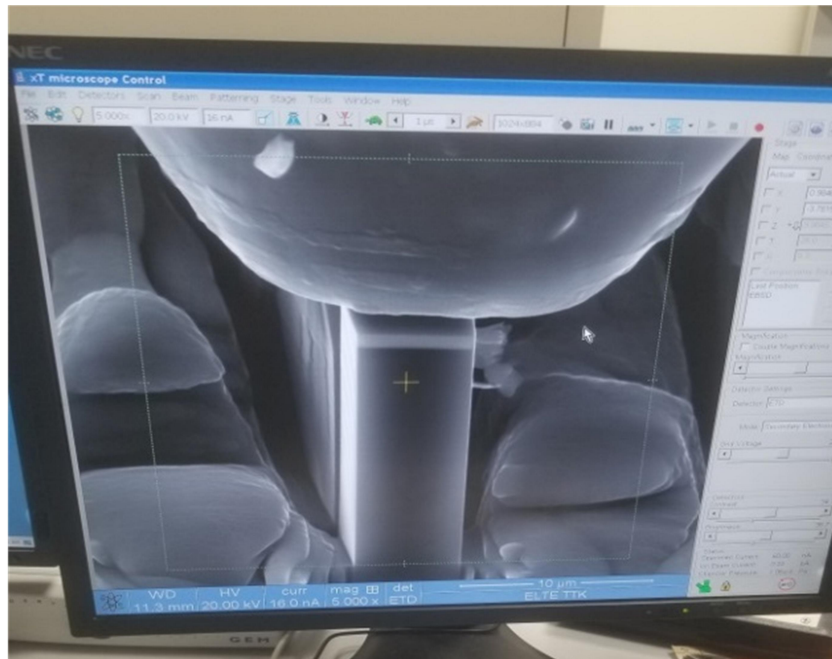


Figure 11: *Starting of Compression process*

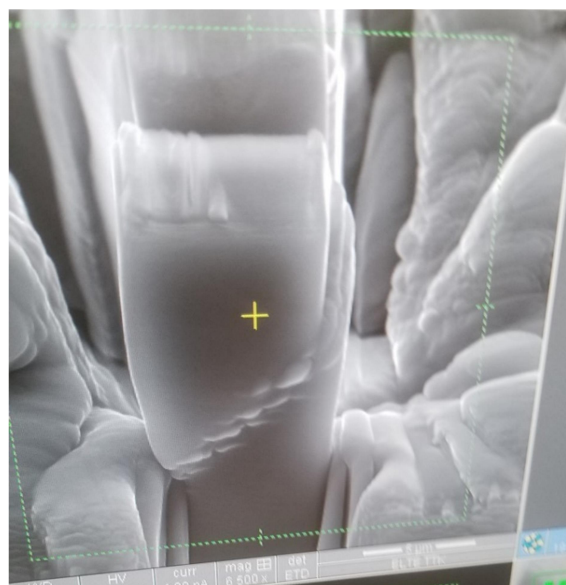


Figure 12: *Pillar after compression test*

During the compression test the following data were recorded by computer program

- Load or force applied
- Time
- Displacement at each load
- AE amplitude
- AE energy

From the recorded data we were able to determine the True stress and True strain that helped us to plot true stress-true strain curve.

3.8. TRUE STRESS-TRUE STRAIN CURVE

In order to deal with large plastic deformation of materials, it is necessary to introduce true stress and true strain occurring in samples during testing. The true stress is defined as applied load divided to actual area of cross section of the sample. This is different from normal stress (σ) which is given by dividing the load applied to the original cross section area of the sample. In compression test if the original length of the sample is l_0 and current length after compression is l then the engineering strain (ε) will be the change in length divided to original length:

$$\varepsilon = \frac{\Delta l}{l_0} \quad (4)$$

The true strain is defined such that its increment $d\varepsilon_T$ is equal to the ratio $\frac{dl}{l}$

After integration the true strain is given as

$$\varepsilon_T = -\ln(1 - \varepsilon) \quad (5)$$

By considering the volume of the sample does not change during the compression test then the true stress σ_T can be calculated as

$$\sigma_T = \sigma \cdot (1 - \varepsilon) \quad (6)$$

3.9. ELECTRON BACKSCATTER DIFFRACTION (EBSD)

This technique is based on the principle that a beam of electrons generated in Scanning Electron Microscope (SEM) is the source of randomly scattered electrons in a specimen

The backscattered electrons that escape the sample generate kikuchi pattern on phosphorous screen which is linked to the specimen crystal structure.

Different crystal orientation provides different patterns hence enable us to study the microstructures, twin boundaries and deformation mechanism. Mostly this technique is full automated, fast and provides accurate, reproducible and statistically meaningful crystallographic orientation.

During the operation of EBSD the incident primary electrons will be scattered within the sample in all directions, high energy electrons which exit the specimen via surface of incidence after one or more scattering events are backscatter electrons

The kikuchi patterns are imaged in SEM, the resolution depends on the accelerating voltage which controls the depth of penetration of electrons in the specimen.

The specimen and phosphor screen are position in the chamber so that a large angular range can be obtained. The shorter distance between the source and pattern center the larger the angular range of EBSP imaged.

When the orientation of the grains change, the structure of the boundary change too due to crystal orientation. This technique is capable to measure thousands of grains boundaries in the materials. The following were some of EBSD maps from our samples.

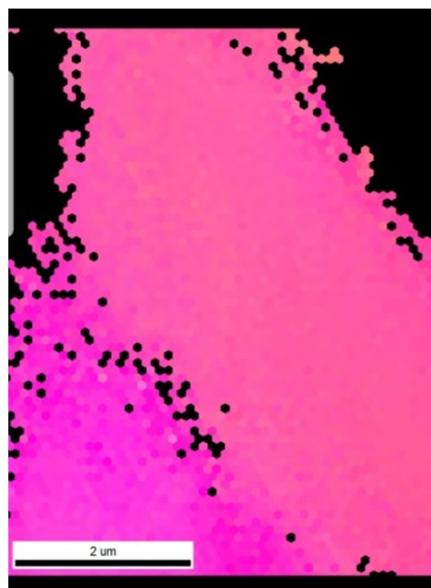


Figure 13: *EBSD mapping for sample loaded at 0.003 μ m/s during full deformation*

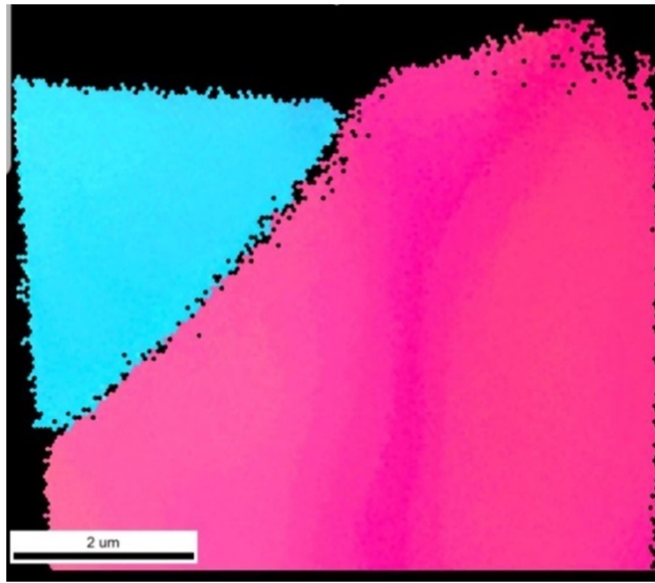


Figure 14: *EBSD mapping for sample loaded at $0.01\mu\text{m/s}$ during full deformation*

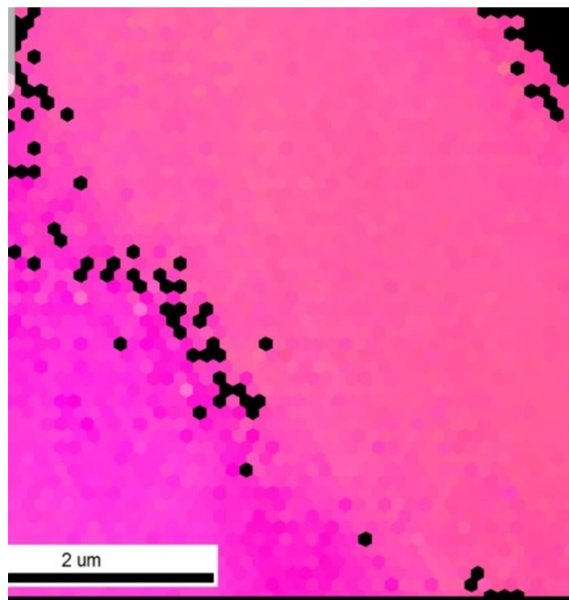


Figure 15: *EBSD mapping for sample loaded at $0.05\mu\text{m/s}$ during full deformation*

4. RESULTS AND DISCUSSION

4.1. Results for indentation tests

Experiment results for all three samples were recorded by computer and the following were results represented in Graphs

Results for high purity Al-3Mg alloy – S₁

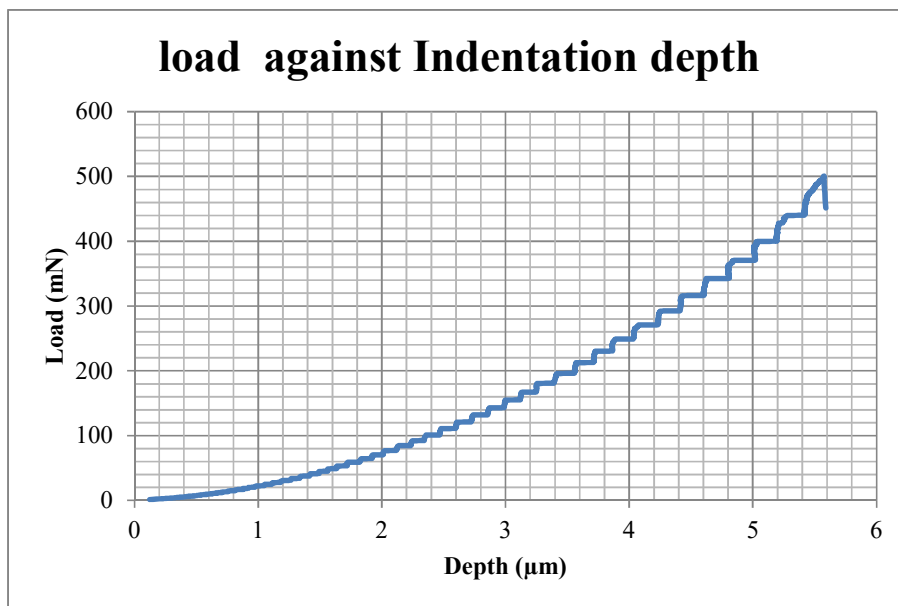


Figure 16: Graph of load-indentation depth for Aluminium-Magnesium Alloy high purity immediately after Quenching

According to the figure 16 it showed that there was plastic instability in sample S₁ as stress drop behavior in steps was observed. The plastic instability observed means that there were the interaction between the dislocations and diffused solute atoms in the material.

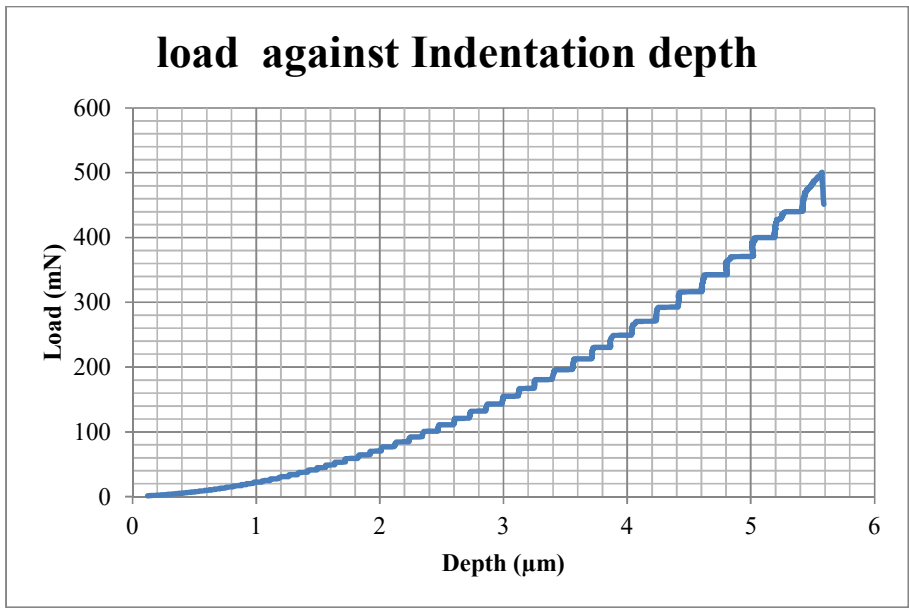


Figure 17: Graph of load-indentation depth for Aluminium-Magnesium Alloy high purity two hours after Quenching

The graph in Figure 17 showed the presence of plastic instability in the sample in two hours later after quenching.

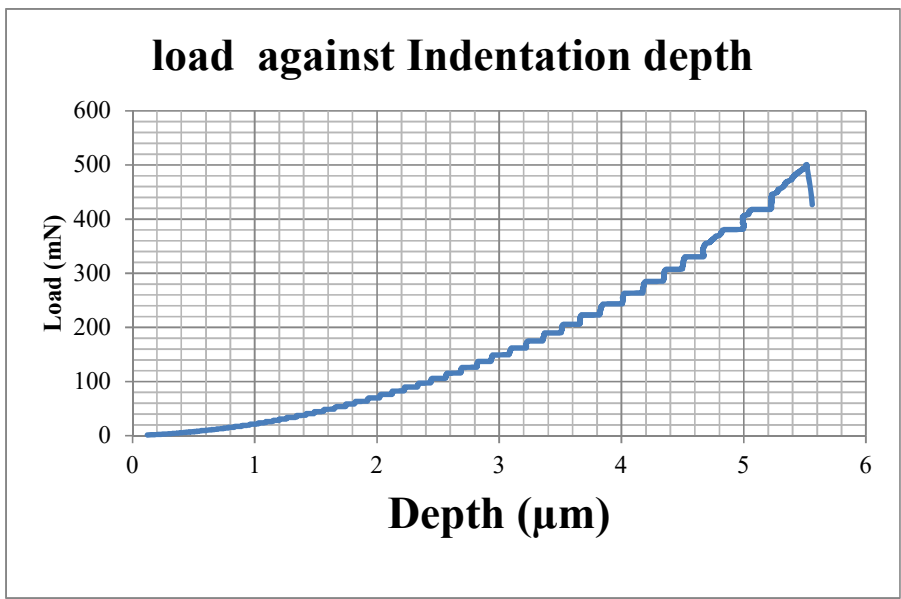


Figure 18: Graph of load-indentation depth for Aluminium-Magnesium Alloy high purity one day after Quenching

Figure 18 also shows the tendency of plastic instability in the material after one day since the sample was quenched

All three graphs in Figures 16, 17 and 18 have the same features even for that of one day after quenching; this indicates in this sample there was no formation of GP zone that could suppress the solute atoms.

In all experiments the maximum load applied was the same and the maximum depth reached were 5.6 μm , 5.6 μm and 5.5 μm in Figures 16, 17 and 18 respectively. This indicates that the hardness in the materials is constant and does not depend on the storing time it remains the same in all three cases. Finally the Plastic instability in Aluminium- Magnesium high purity was very stable.

Results for commercial purity Al-3Mg alloy – S₂

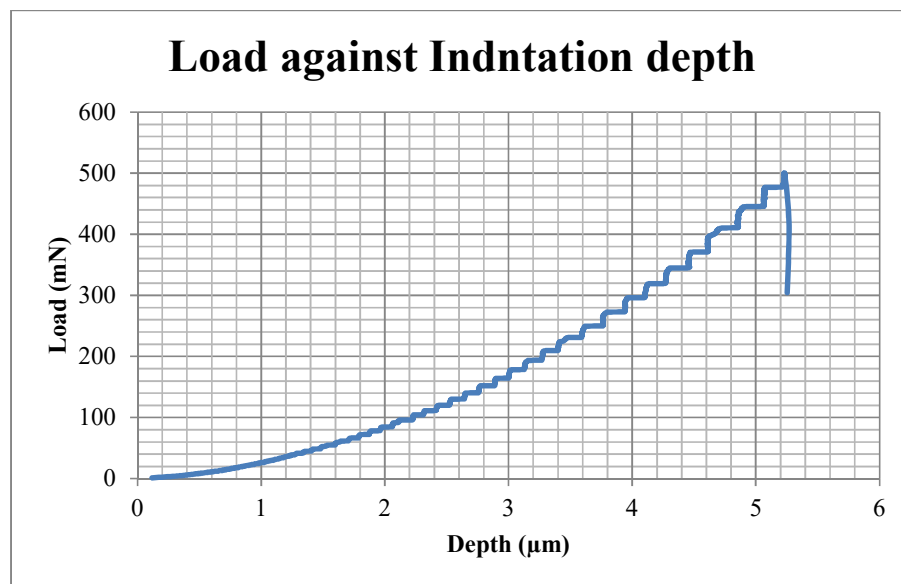


Figure 19: Graph of load-indentation depth for Aluminium-Magnesium Alloy commercial purity immediately after Quenching

In figure19 it showed that there was plastic instability in sample S₂ as stress drop was observed. The plastic instability which observed means that there was an interaction between the dislocations and diffused solute atoms in the material immediately after quenching.

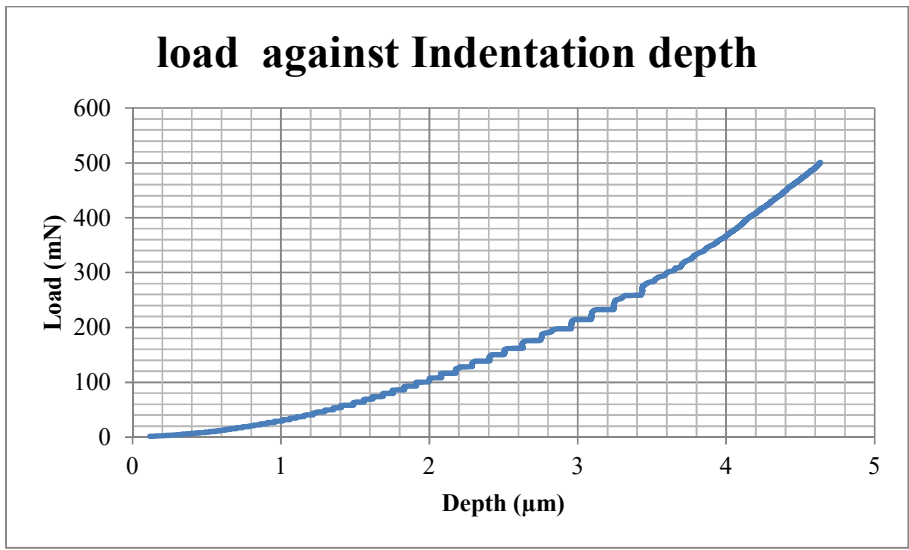


Figure 20: Graph of load-indentation depth for Aluminium-Magnesium Alloy commercial purity of two hours after Quenching

In the figure 20 above it showed that at the beginning there was a tendency of plastic instability in the sample after sometimes it disappeared, that is was sign for precipitation starting that lead the decrease in number for solute atoms and increase in solute atom spacing hence the interaction between the solute atoms with dislocations decreased.

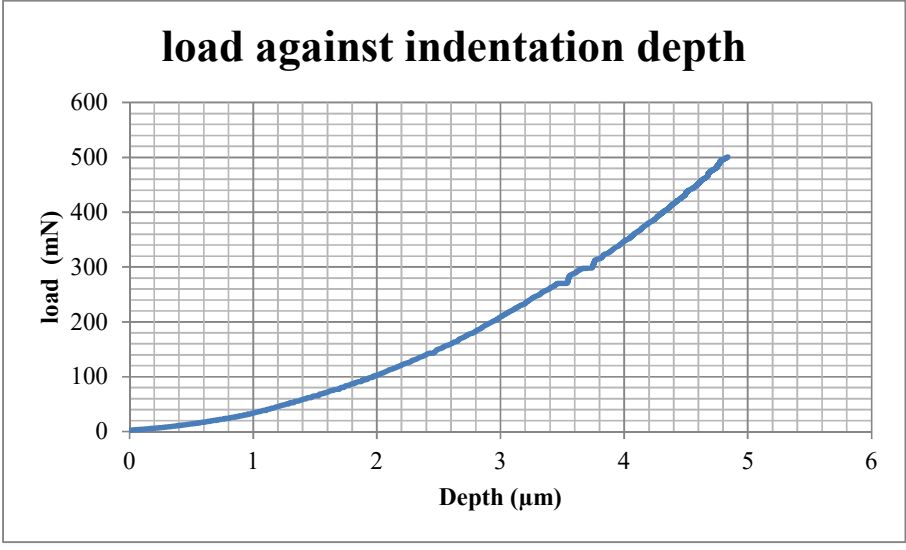


Figure 21: Graph of load-indentation depth for Aluminium-Magnesium Alloy commercial purity of one day after Quenching

In the figure 21 there was no plastic instability shown in the sample just one day after quenching, this shows that precipitation in the sample was completed, and the hardness increased in this sample. The plastic instability in Aluminium –Magnesium commercial was not stable like

that of Aluminium-Magnesium purity because it was not too homogeneous due to presence of impurities

Comparing Hardness between Al-Mg purity and Al-Mg commercial

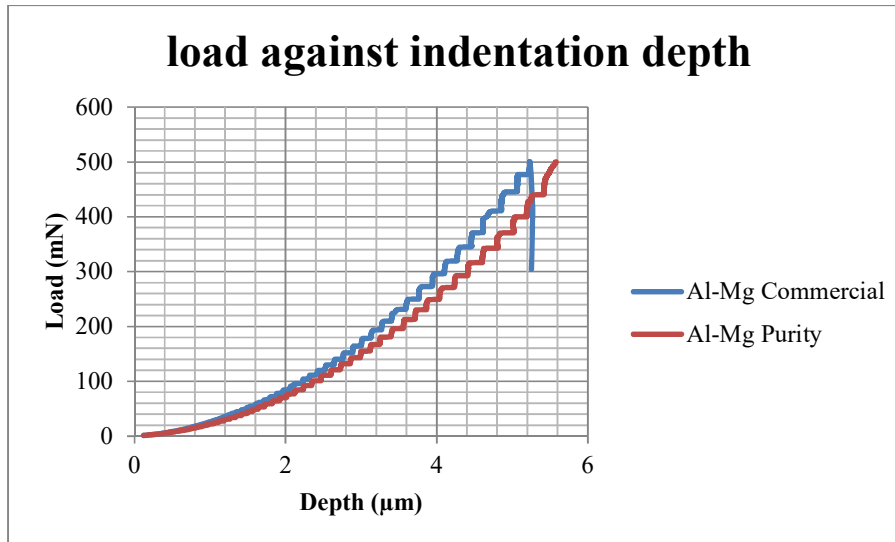


Figure 22: Graph for Al-Mg purity and Al-Mg commercial immediately after quenching

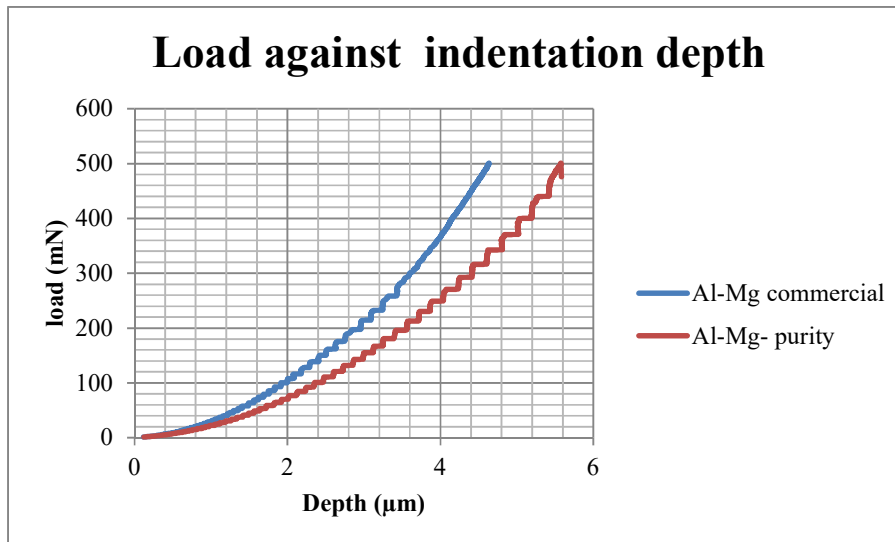


Figure 23: Graph for Al-Mg purity and Al-Mg commercial two hours after quenching

From the graphs in figure 22 and 23 above the indentation depth of purity alloy is higher than that of commercial alloys that indicate that the Hardness values for commercial are higher than that of purity Alloys(the lower depth of penetration the harder the material)

Results for Al-Zn-Mg-Zr alloy – S₃

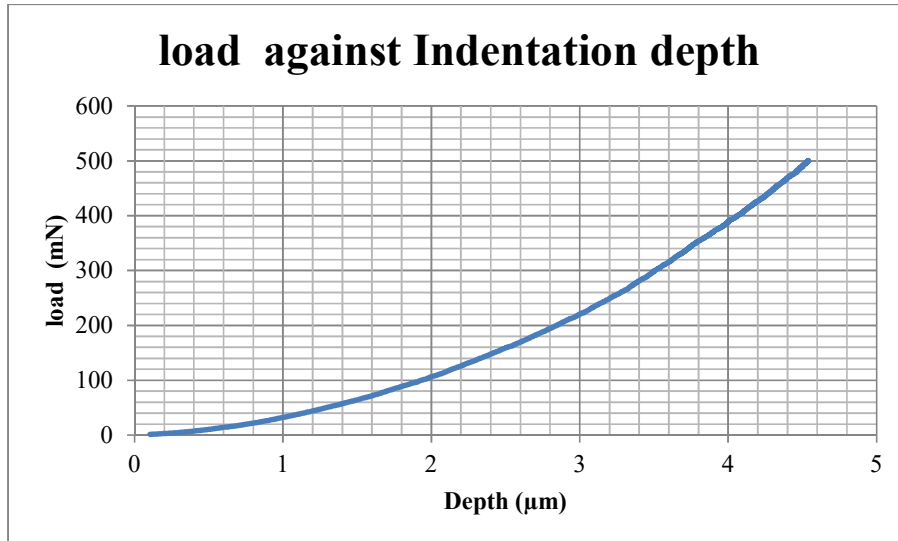


Figure 24: *Graph of load-indentation depth for Aluminium-Zinc- Magnesium-zircon immediately after Quenching*

In the figure 24 above there was no any tendency of plastic instability whilst the sample was tested immediately after quenching. This was due to rapidly formation of GP zone which suppress the solute atoms hence there were no plastic deformation. By this incident we can say the material had high solute content that led the rapidly formation of GP zone.

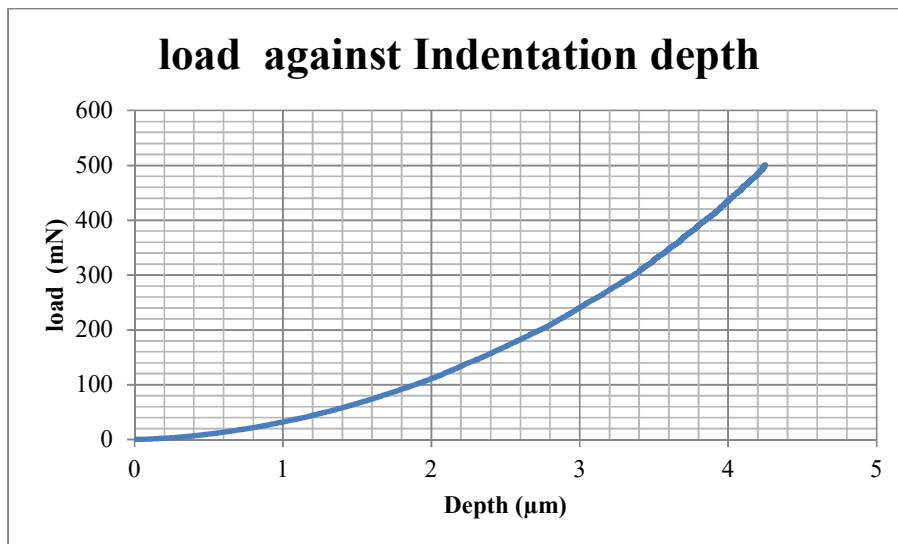


Figure 25: *.Graph of load-indentation depth for Aluminium-Zinc- Magnesium-zircon two hours after Quenching*

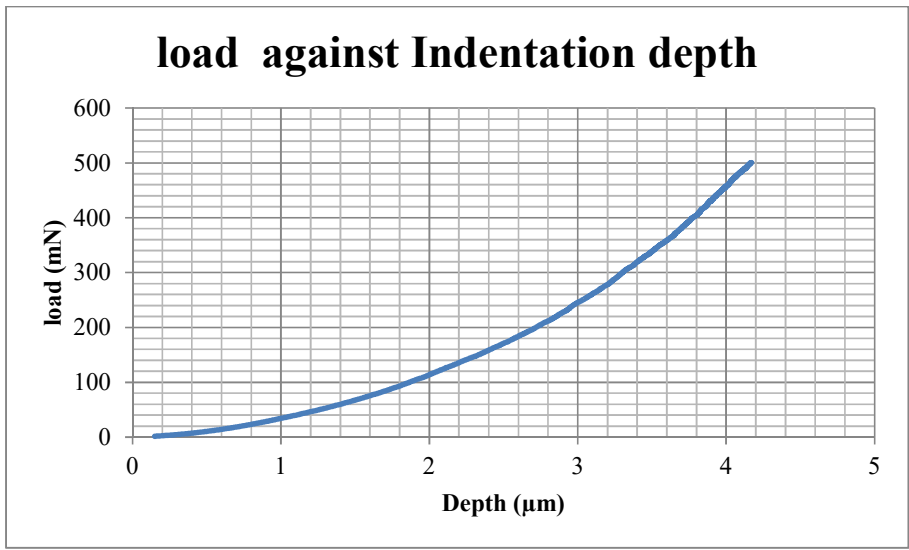


Figure 26: Graph of load-indentation depth for Aluminium-Zinc- Magnesium-zircon one day after Quenching

Results for both figures 25 and 26 were the same due to early rapidly GP zone formation in the sample which suppress the solute atoms hence there were no plastic instability in the sample. The indentation values showed in figures 16.17 and 18 are continuously decreasing from 4.5μm, 4.2μm and 4.1μm for immediately, two hours and one day after quenching respectively. This indicates the increase in Hardness as time goes after quenching.

Hardness comparison in all three samples

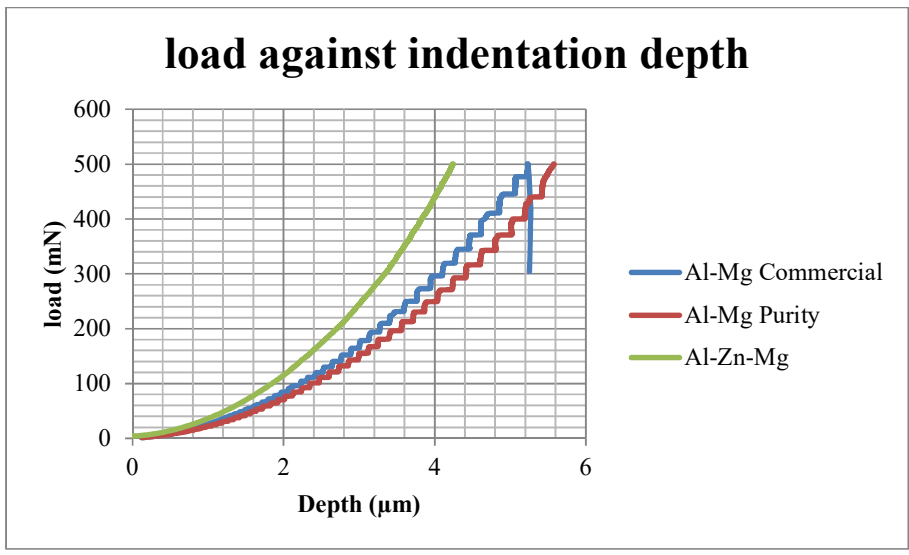


Figure 27: Graph for Al-Mg purity, Al-Mg commercial and Al-Zn-Mg immediately after quenching

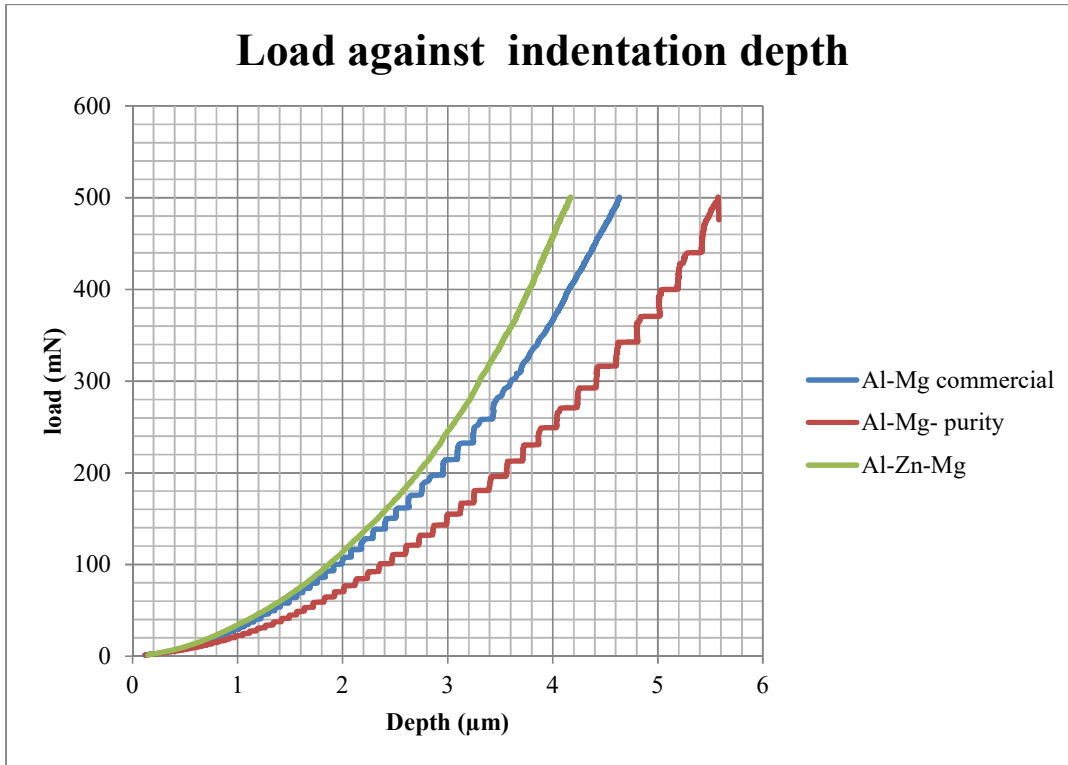


Figure 28: Graph for Al-Mg purity, Al-Mg commercial and Al-Zn-Mg two hours after quenching

In figures 27 and 28 above it shows that the Al-Zn-Mg has the lowest indentation depth hence the hardest to all, this indicates the formation of GP zones in the materials increases the hardness of the alloys.

4.2. RESULTS FOR MICRO PILLARS COMPRESSION TESTS

Depending on the results shown in Indentation test showed that the Aluminium-Magnesium purity showed good behavior in plastic instability so I decided to check its instability behavior by micro pillars compression tests with help of Acoustic Emission (AE) technique. The results were as shown

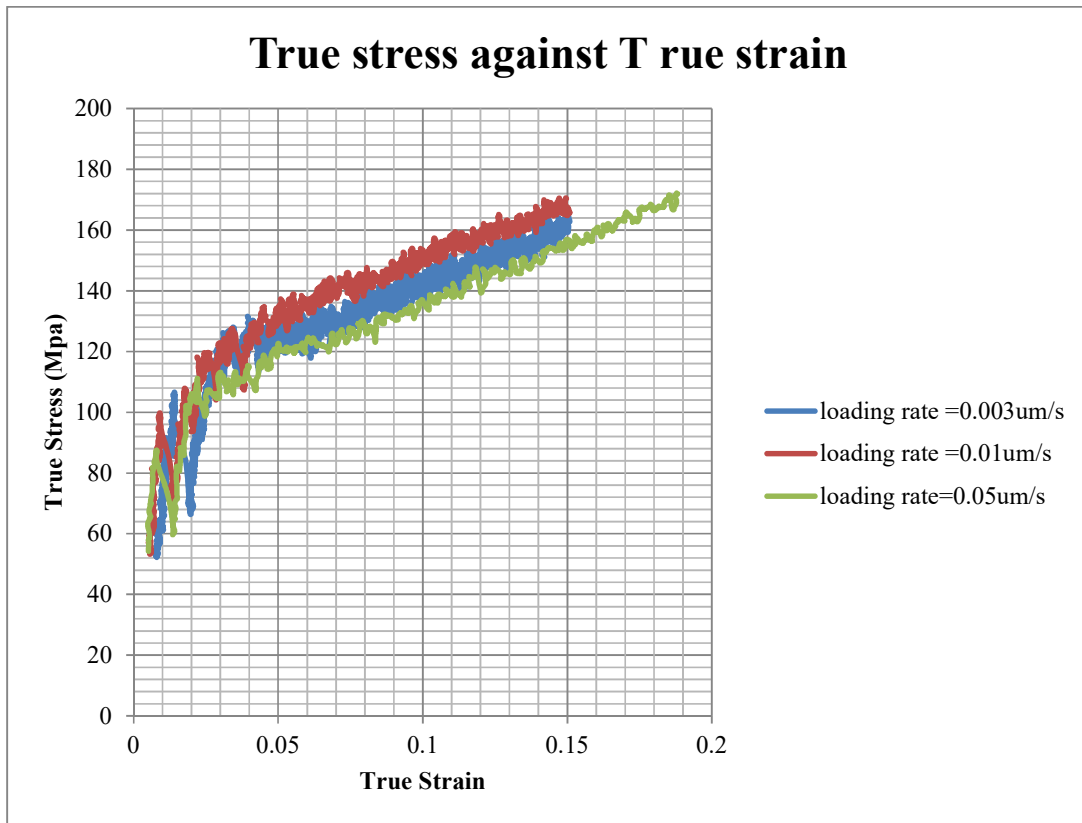


Figure 29: *The graph of True stress –True strain at different loading rate for 20% deformed samples*

From the graph in figure 29 above the nature were the same for all three graphs; they have approximately values for proof maximum stresses. By these results we can say that the loading rate had no effect on these samples (it was global behavior)

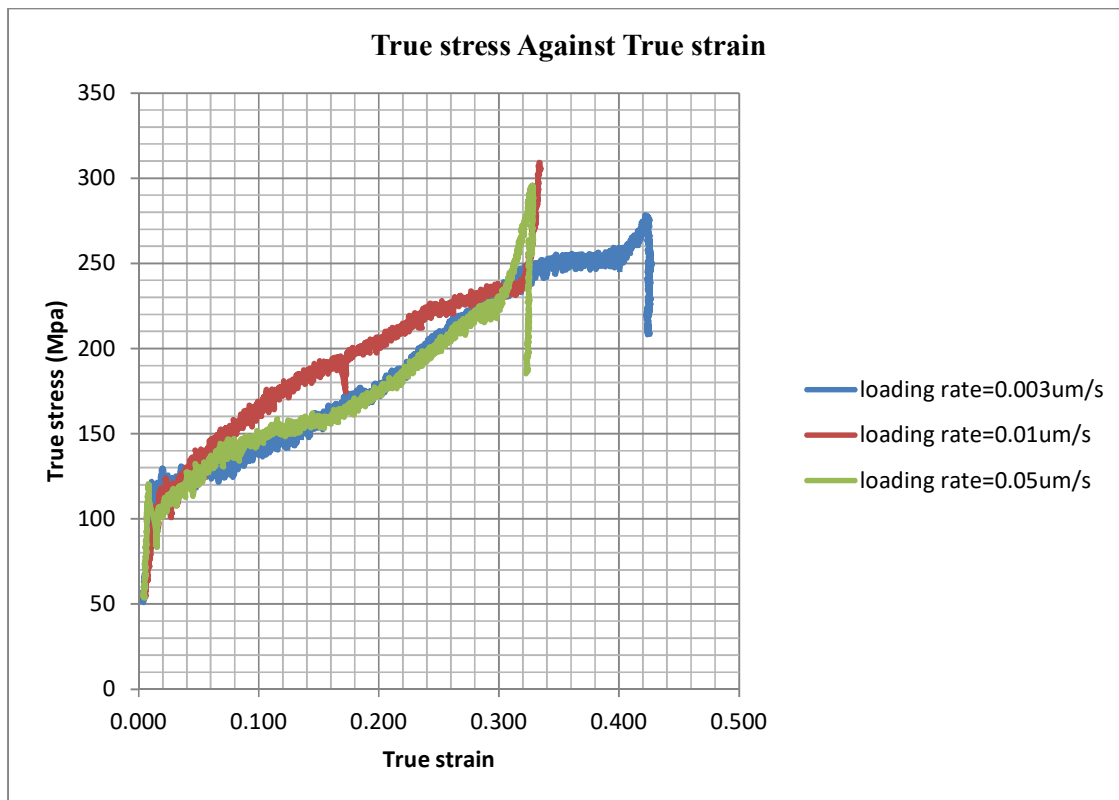


Figure 30: *The graph of True stress –True strain at different loading rate for full deformed samples*

From the graph in figure 30 above, two samples had the same behavior as they have the same approximately values for proof stress and maximum stresses but one had higher value for maximum stress than others. This effect cannot be counted as an effect of loading rate because that of higher loading rate is similar to that of lowest loading rate. The diversion occurred to that whose loading rate was in between.

Due to that I decided to plot other graphs by shifting the graphs so as to be able to identify any difference as shown in the figure 31 below

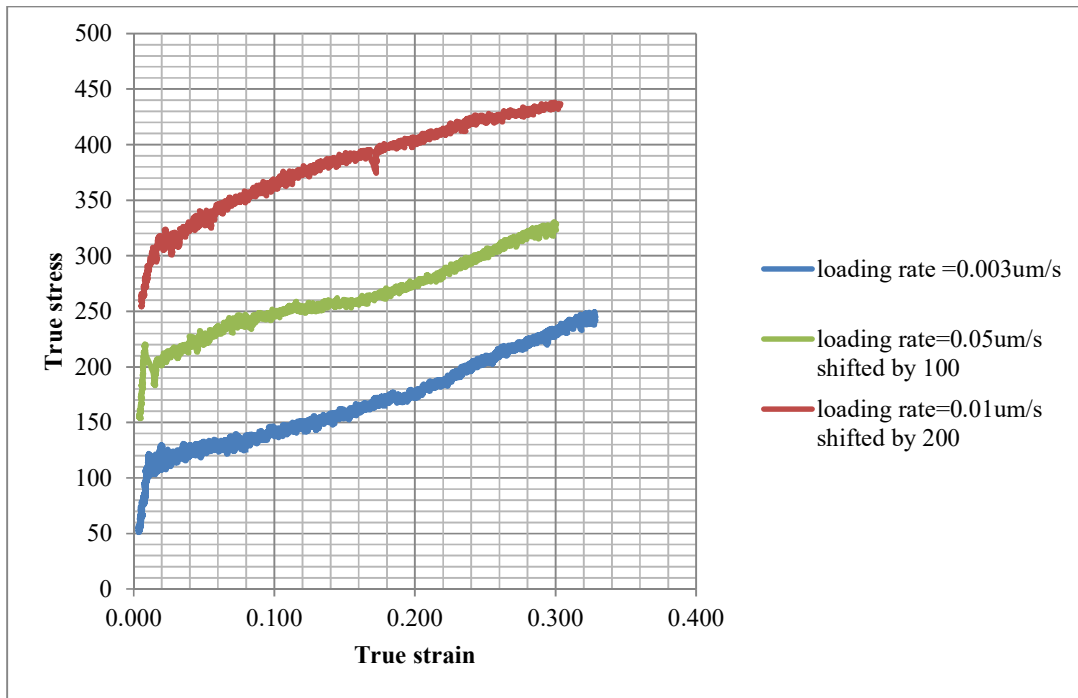


Figure 31: The graph of True stress – True strain at different loading rate for full deformed samples after shifting graphs so as to separate them

After separated the graphs in the figure 31 above, the first two graphs from the bottom showed linear behavior after proofing stress point, thus we can say at that region the stress was directly proportional to strain ($\sigma_T \propto \varepsilon_T$) but the other one showed some features like curve, from that we can term it as local deformation behavior where the stress is related to a power function of the strain, $\sigma_T \propto \varepsilon_T^\alpha$ with the exponent, α .

After doing EBSD mapping, we realized that the sample with high stress was due to grain boundary which act as an obstacle to dislocation movement hence cause the rise in stress than other two values. The grain boundary was realized due to two sides to have different grains orientations (which represented for different colors in EBSD mappings). That pillar with grain boundary deformed like poly crystal while others deformed as single crystal.

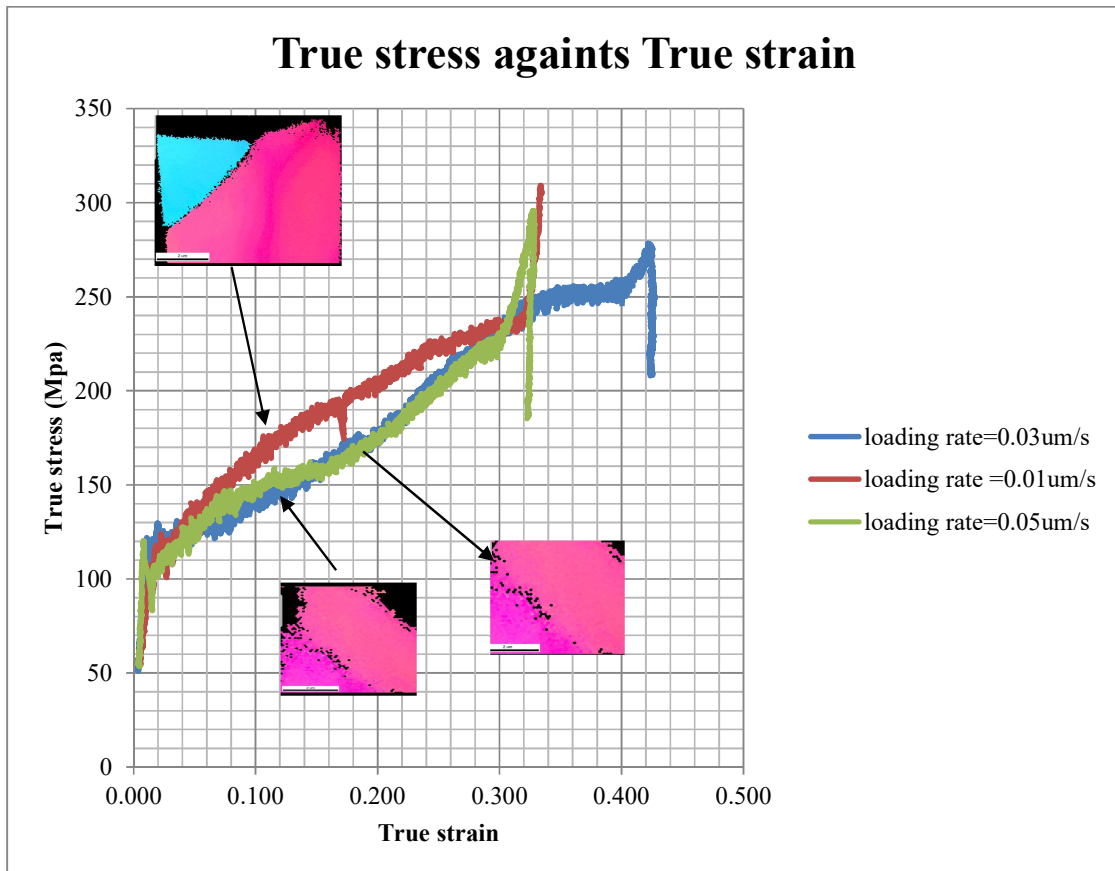


Figure 32: *The graph of True stress –True strain at different loading rate for full deformed samples with their EBSD maps*

The conditions of Full deformed and that of 20% deformed were also compared as shown in figures 33 and 34 below

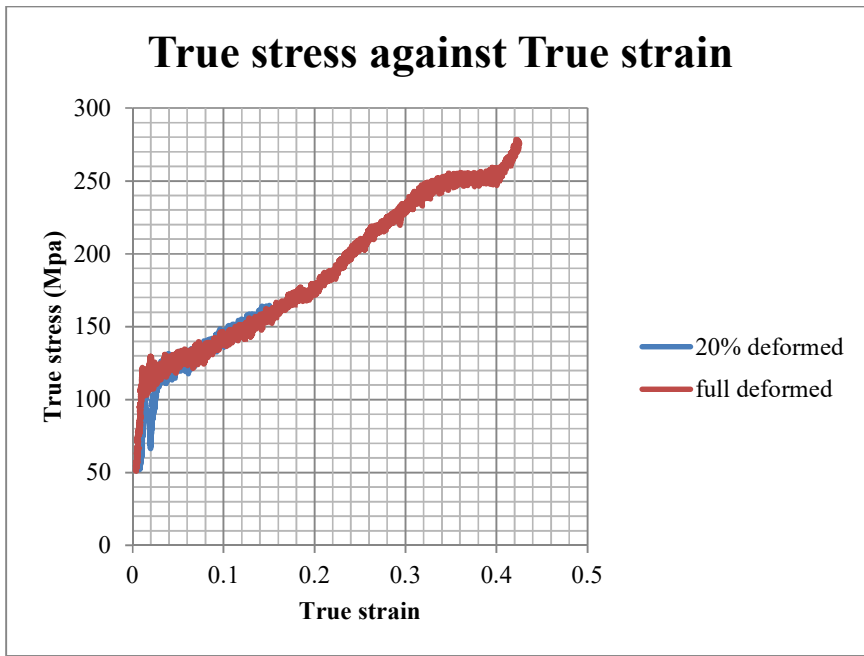


Figure 33: The graph of True stress-True strain at loading rate of $0.003\mu\text{m/s}$ for full and 20% deformed samples.

From the figure 33 above we observed that the Global behavior during deformation was well produced in both samples as both curves behaves similar up to 20% deformation.

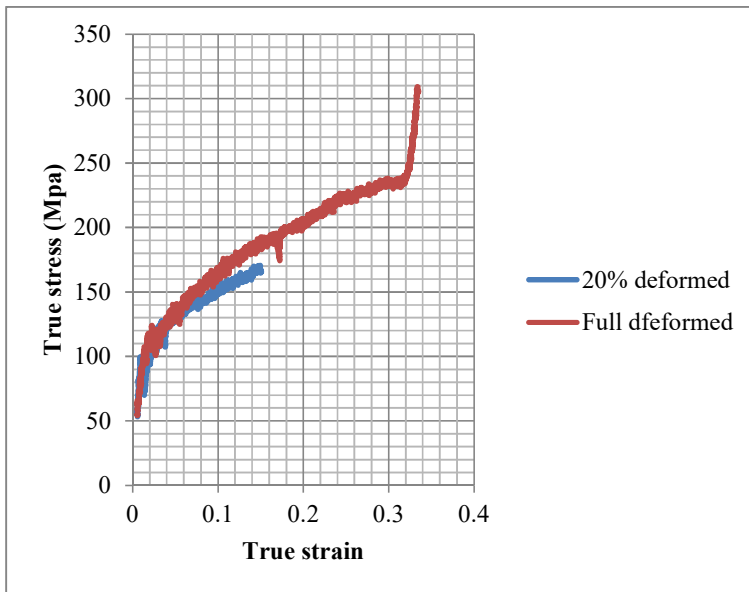


Figure 34: The graph of True stress-True strain at loading rate of $0.003\mu\text{m/s}$ for full and 20% deformed samples.

In the figure 34 one sample under go Global deformation while the other was in local deformation hence their nature was not similar.

The results for AE signals and stresses were as shown below

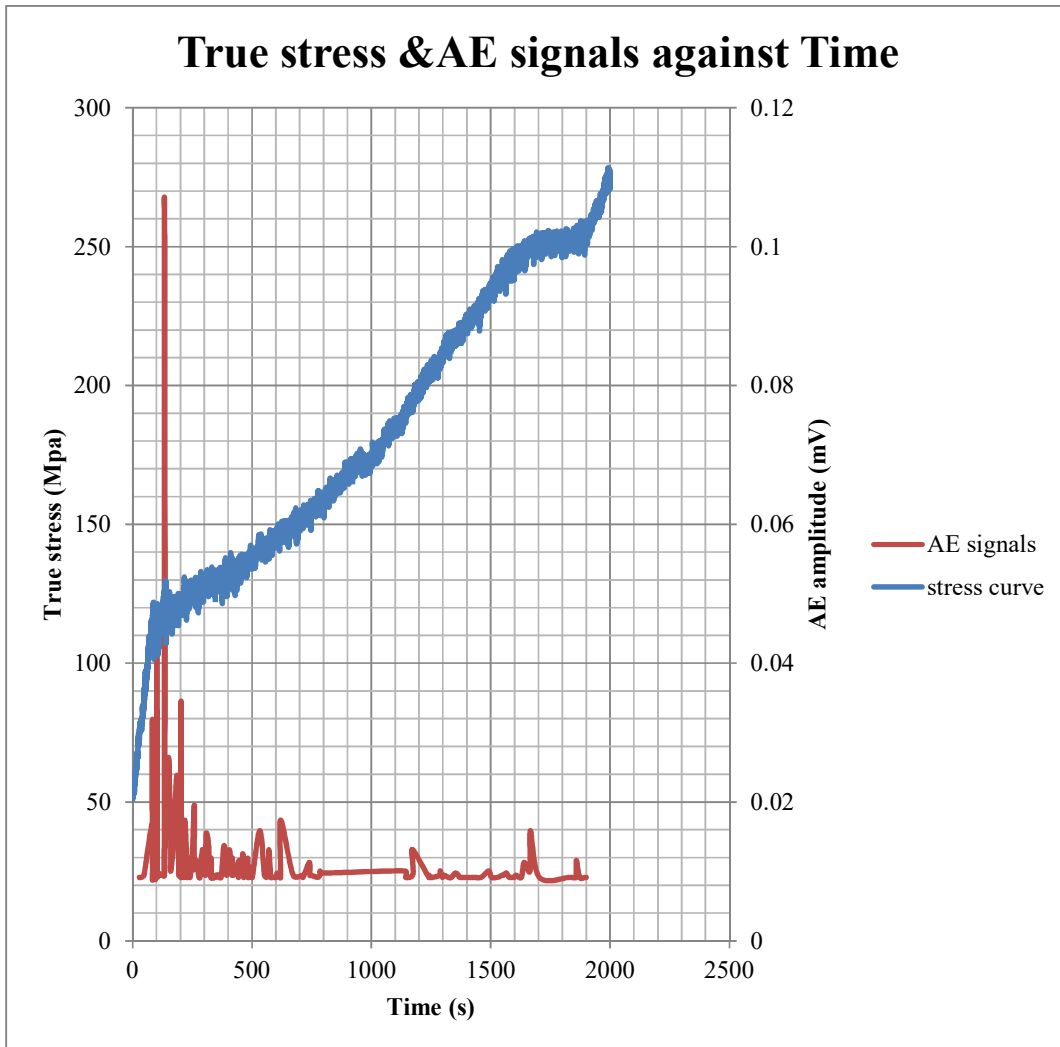


Figure 35: The graph for relation of AE signals and Stresses for full deformed sample at $0.003\mu\text{m/s}$

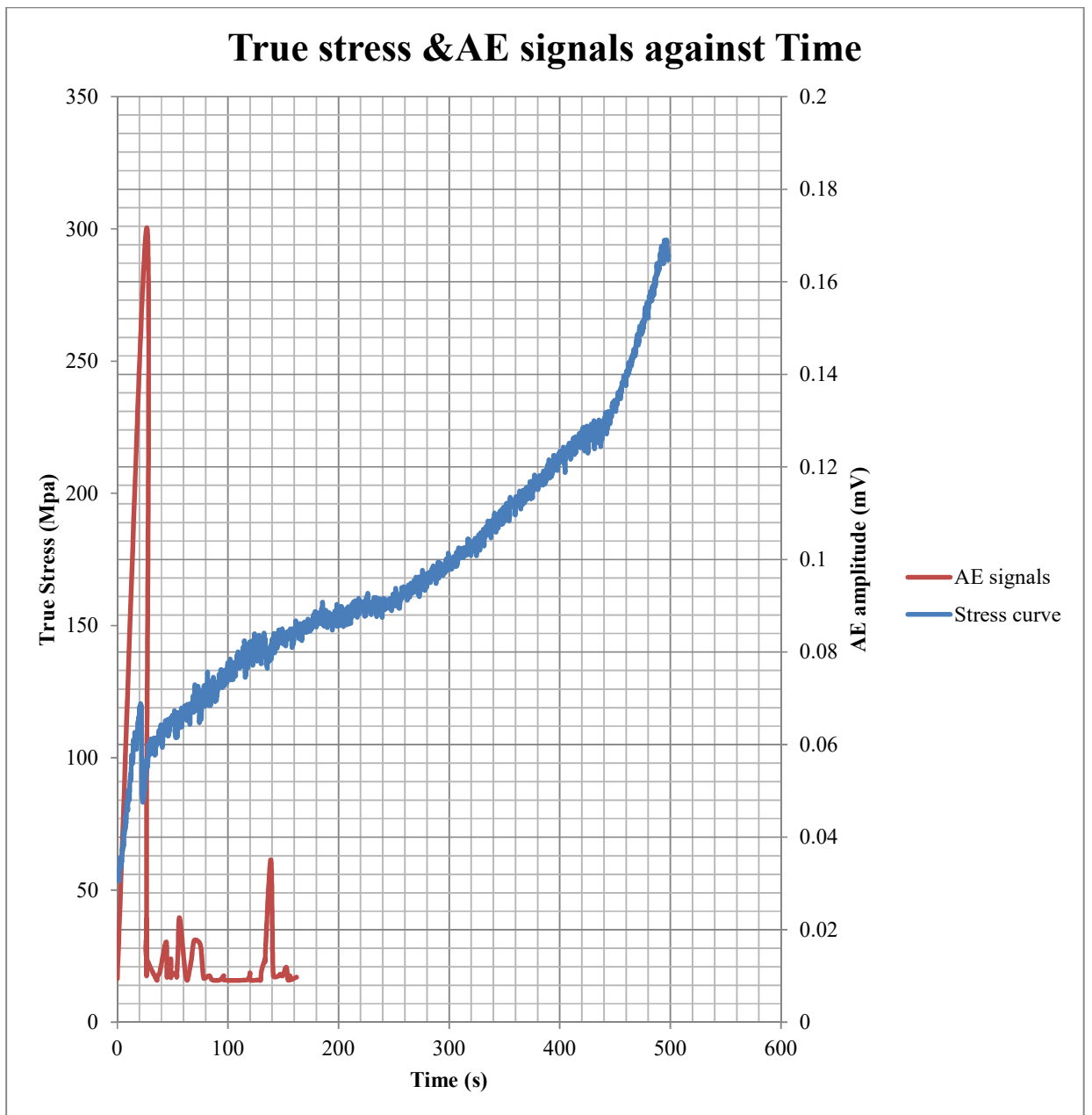


Figure 36: The graph for relation of AE signals and Stresses for full deformed sample at $0.01\mu\text{m/s}$

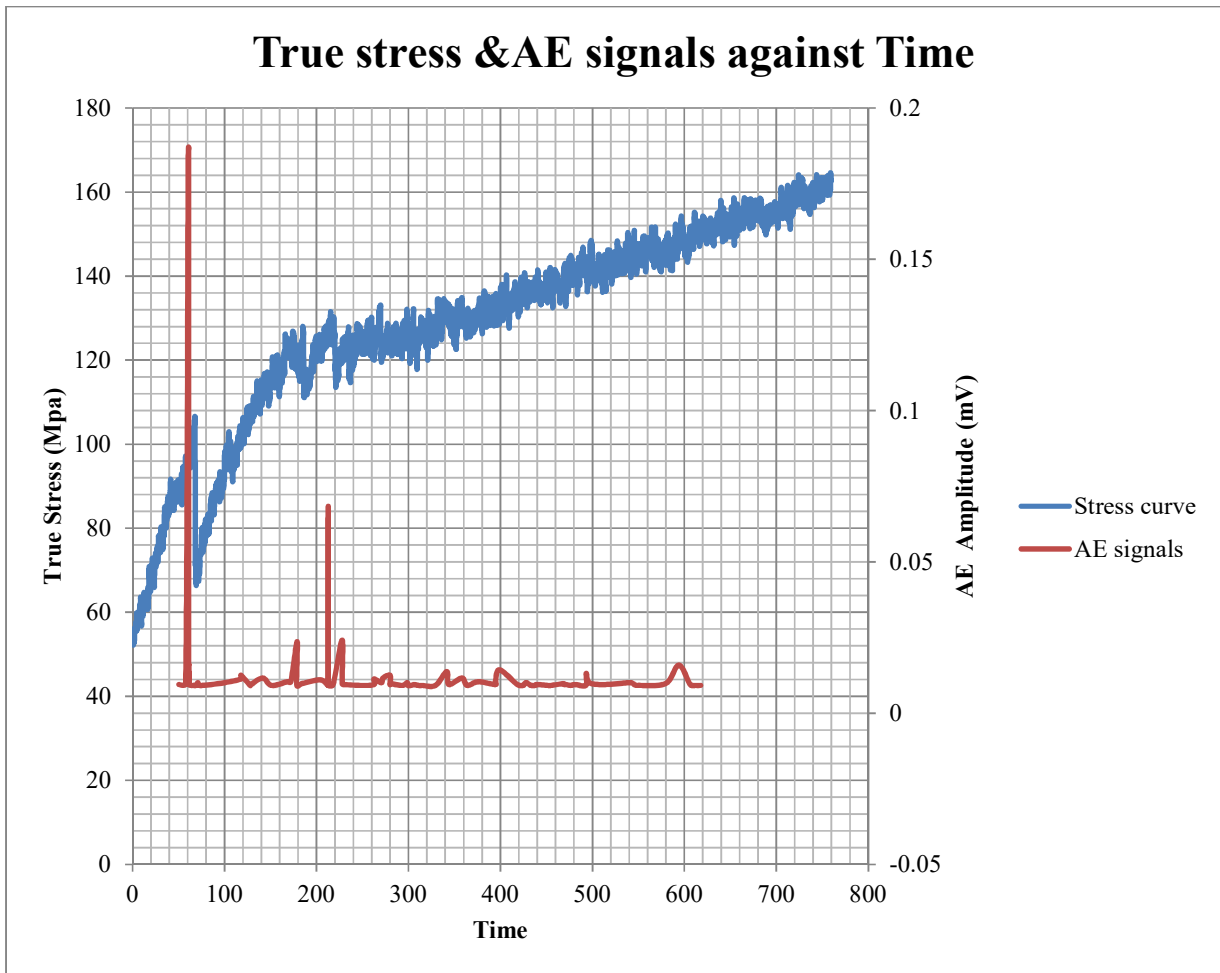


Figure 37: The graph for relation of AE signals and Stresses for 20% deformed sample at $0.003\mu\text{m/s}$

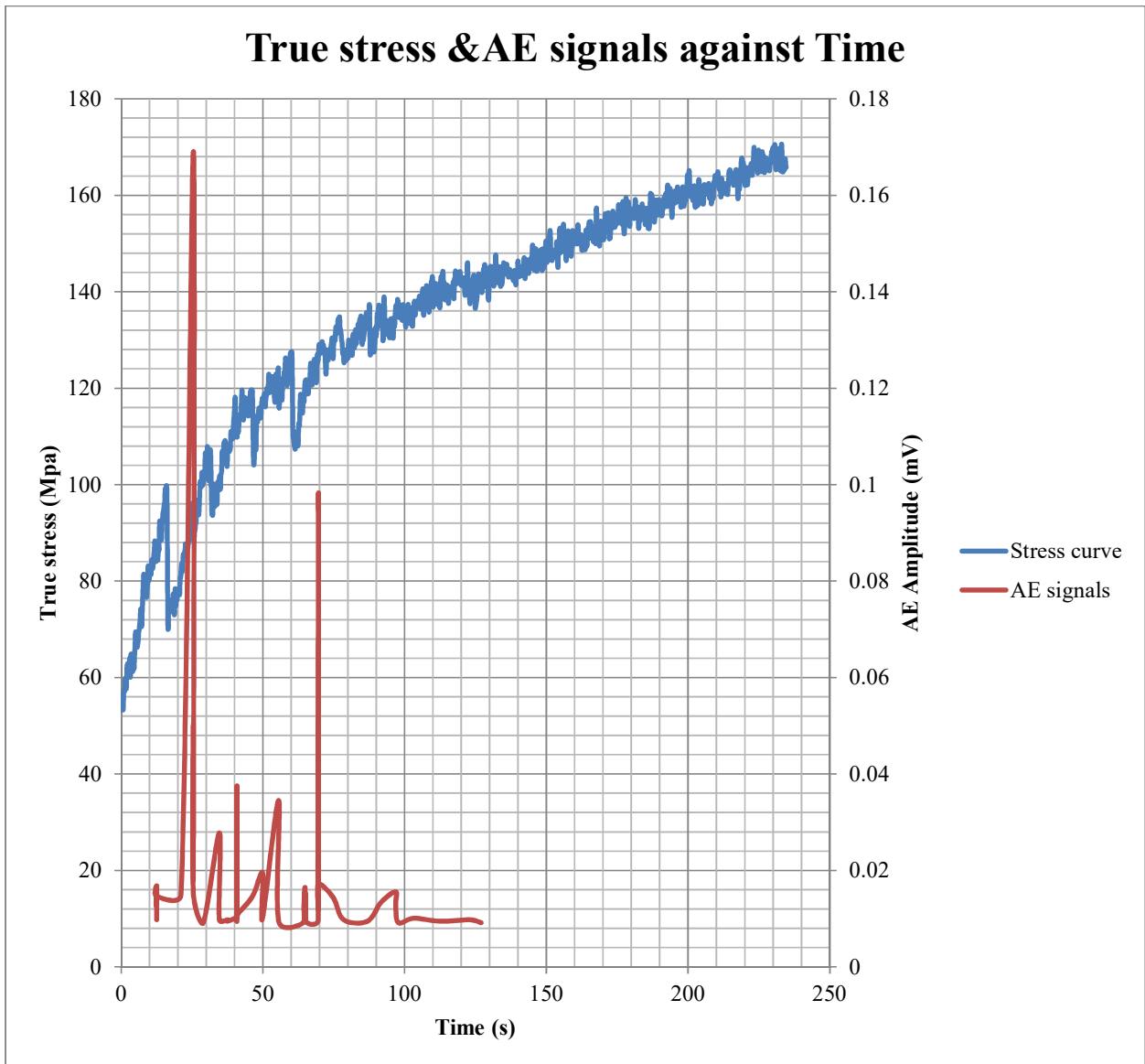


Figure 38: The graph for relation of AE signals and Stresses for 20% deformed sample at $0.01\mu\text{m/s}$

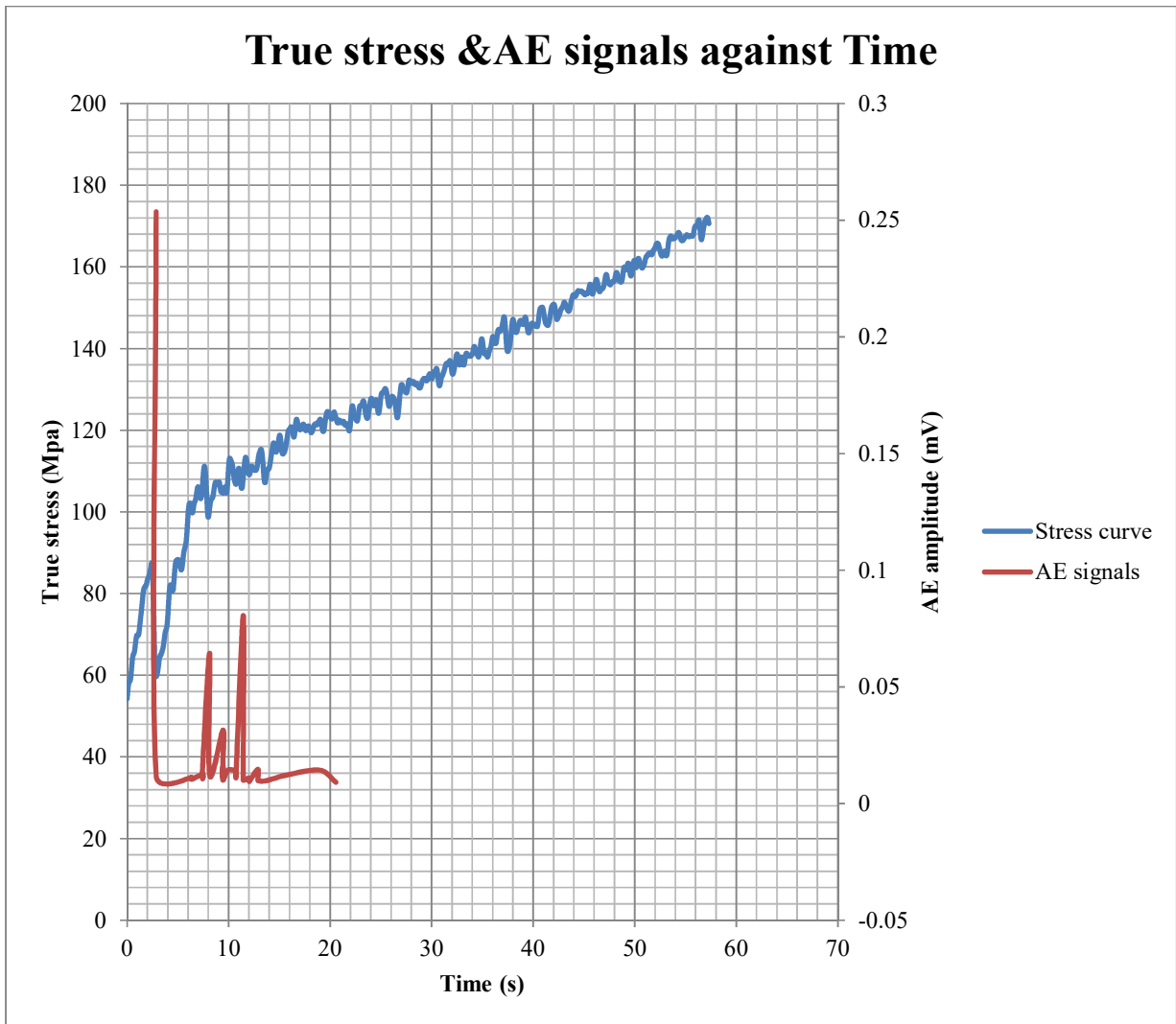


Figure 39: *The graph for relation of AE signals and Stresses for 20% deformed sample at 0.05 μ m/s*

In the figures 35,36,37,38 and 39 all showed that when the stress drop is larger the AE signals were also high, because the AE signals are transient elastic waves which generated by rapid release of energy from localized sources within the materials. When the structures change suddenly like collective dislocations, break out of dislocations the AE signals are also detected. Therefore for any dynamic phenomena takes place in the materials during plastic deformation can be noticed through these AE signals.

5. CONCLUSION

In my diploma work I have investigated the phenomenon of plastic instabilities in three alloys: high purity Al-3Mg, commercial purity Al-3Mg and Al-4.8Mg-1.2Zn-0.14Zr, which are basic materials in the Al industry. The investigations were carried out by using indentation, micro pillar-compression, scanning electron microscopy and focused ion-beam (SEM/FIB) measurements, as well as acoustic emission (AE) technique. The main results are the followings:

1) The experimental results have shown that the high purity Al-based Al-3Mg alloy is a stable solid solution. This sample always shows plastic instability, and its hardness is constant in all tests during storing at room temperature. The commercial purity Al- commercial purity showed the plastic instability but was not stable. The phenomenon of plastic instabilities taking place in this sample disappeared in the test carried out after storing the sample at room temperature for some days. Furthermore, its hardness was increasing during storing at Room Temperature (RT).

2) In the case of Al-Zn-Mg-Zr alloy, plastic instability was not observed at all due to formation of Guinier-Preston (GP) zones which formed rapidly in the material and suppressed the solute atoms. Because of the effect of GP zones, the hardness of this alloy is continuously increasing during storing at room temperature, and it was very hard compared to other to alloys.

3) In the case of the stable solid solution high purity Al-3Mg alloy the phenomenon of plastic instabilities was investigated by micro pillar compression test, which was combined with the acoustic emission technique. It was shown that in spite of the same global behavior, the local behavior characterizing plastic instabilities is depending on the velocity of the deformation process.

4) Results of the complex investigation including micro-pillar compression and acoustic emission technique have shown that the stress drops describing plastic instabilities can be related to the AE signals. In the case of single crystal micro-pillars, the AE signals were proportional to stress drop in all pillars, when the stress drop was larger the AE signals were high too. This relationship can be effect by the present of grain boundaries in the sample.

6. FURTHER STUDIES

Through this work we have seen the relationship between the stress drops with AE signals, wherever there is higher stress drop there is higher AE signals. For further studies we should do quantitative analysis and find out the mathematical relation between stress drop and AE signals in Aluminium alloys as our case study.

Moreover, we will focus to study in details the mechanisms of plastic instabilities in Aluminium alloys by starting with mostly and widely used alloys

REFERENCES

1. CHINH, N., CSIKOR, F., KOVÁCS, Z. & LENDVAI, J. (2000). *Critical concentration of Mg addition for plastic instabilities in Al–Mg alloys*. *J Mater Res* 15(5), 1037–1040.
2. N.Q. Chinh, J. Gubicza, Zs. Kova'cs, and J. Lendvai Department of General Physics, Eo'tvös University, Budapest, 1117 Budapest, Hungary *Depth-sensing indentation tests in studying plastic instabilities*
3. Burton, M.S, (1956). *Applied Metallurgy for Engineers*
4. DOBROŇ, P., CHMELÍK, F., BOHLEN, J., HANTZSCHE, K., LETZIG, D. & ULRICH KAINER, K. (2009). *Acoustic emission study of the mechanical anisotropy of the extruded AZ31 alloy*. *Int J Mater Res* 100(6), 888–891.
5. GUBICZA, J., CHINH, N.Q., HORITA, Z. & LANGDON, T. (2004). *Effect of Mg addition on microstructure and mechanical properties of aluminum*. *Mater Sci Eng A* 387, 55
6. Rollason, E.C, (1973). *Metallurgy for Engineers*
7. HÜTSCH, J. & LILLEODDEN, E.T. (2014). *The influence of focused ion beam preparation technique on microcompression investigations: Lathe vs. annular milling*. *Scr Mater* 77, 49–51.
8. ISHITANI, T., UMEMURA, K., OHNISHI, T., YAGUCHI, T. & KAMINO, T. (2004). *Improvements in performance of focused ion beam cross-sectioning: Aspects of ion-sample interaction*. *J Electron Microsc* 53(5), 443–449.
9. SCRUBY, C., WADLEY, H., RUSBRIDGE, K. & STOCKHAM-JONES, D. (1981). *Influence of microstructure on acoustic emission during deformation of aluminium alloys*. *Metal Sci* 15(11–12), 599–608
10. Kristián Máthis and František Chmelík Department of Physics of Materials, Faculty of Mathematics and Physics, Charles University, Prague, Czech Republic *Exploring Plastic Deformation of Metallic Materials by the Acoustic Emission Technique*
11. N.Q. Chinh, F. Csikor, Zs. Kovács, and J. Lendvai, *J. Mater. Res.* 15, 1037 (2000).
12. *Microstructure and Properties of Aluminum-Containing Refractory High-Entropy Alloys*, O.N. SENKOV, C. WOODWARD, and D.B. MIRACLE, *JOM*, Vol. 66, No. 10, 2014, pages: 2030-204
13. D. Tabor, *Microindentation Techniques in Materials Science and Engineering*, ASTM STP 889, edited by P.J. Blau and B.R. Lawn (American Society for Testing and Materials, Philadelphia, PA, 1986), p. 129.

14. *Kristián Máthis and František Chmelík (2012). Exploring Plastic Deformation of Metallic Materials by the Acoustic Emission Technique, Acoustic Emission, Dr. Wojciech Sikorski (Ed.), ISBN: 978-953-51-0056-0*
15. *N.Q. Chinh, F. Csikor, Gy. Bérces, and J. Lendvai, Alum. Alloys 2, 979 (1998)*
16. *Ask land (1992), Science of engineering materials*
17. *Shackelford, J.F (2000), introduction to material science Of engineers. Fifth edition*
18. *Massalski, T.B., 1989. Phase diagrams in materials science. Metall. Trans. A 20A, 1295-1323*
19. *Cantor, B., 2007. Stable and metastable multicomponent alloys. Ann. Chim. Sci. Mat. 32, 245-256.*

STATEMENT

Name : CHARLES WAMBURA NYAKETOLEKA

ELTE Faculty of Science, Field of studies: Materials Science

Title of the thesis: Investigation of plastic instabilities in Aluminium Alloys

I hereby declare as the author of this thesis, that it is a product of my own and that it contains my own ideas. I used the standard rules for references and quotations consistently. I never used other people's idea without proper reference.

Budapest, 4th January 2021

Charles W
Signature

