Nanoindentation Study of Pure Magnesium

A Thesis Submitted in Partial Fulfillment of the Requirements

for the Degree of

Bachelor of Technology

In

Metallurgical and Materials Engineering

By

Brahmananda Hota (111MM0363)

Pravat Kumar Sahoo (111MM0379)

Nabodit Patra (111MM0576)

Department of Metallurgical and Materials Engineering

National Institute of Technology

Rourkela

2015

Nanoindentation Study of Pure Magnesium

A Thesis Submitted in Partial Fulfilment of the Requirements

for the Degree of

Bachelor of Technology

In

Metallurgical and Materials Engineering

By

Brahmananda Hota (111MM0363)

Pravat Kumar Sahoo (111MM0379)

Nabodit Patra (111MM0576)

Under the Guidance of

Prof. S. K. Sahoo and Prof. S. C. Mishra

Department of Metallurgical and Materials Engineering

National Institute of Technology

Rourkela

2015

National Institute of Technology
Rourkela

Certificate

This is to certify that the thesis entitled, " **Nanoindentation Study of Pure Magnesium”** submitted by **Brahmananda Hota (111MM0363)**, **Pravat Kumar Sahoo** (**111MM0379**) and **Nabodit Patra (111MM0576)** in partial fulfilment of the requirements for the award of **Bachelor of Technology Degree** in **Metallurgical and Materials Engineering** at National Institute of Technology, Rourkela is an authentic work carried out by them under my supervision and guidance.

To the best of my knowledge, the matter embodied in the thesis has not been submitted to any other University/Institute for the award of any Degree or Diploma.

Date: 06/May/2015
Place: Rourkela

Prof. S. K. Sahoo and Prof. S. C. Mishra
Dept. of Metallurgical and Materials Engineering
National Institute of Technology
Rourkela-769008

ACKNOWLEDGEMENT

Foremost, we would like to express our sincere gratitude to our supervisor Prof. Santosh Kumar Sahoo for enlightening us the first glance of research and for his patience, motivation, enthusiasm, and immense knowledge. His guidance helped us in the time of research and writing of this thesis. We could not have imagined having a better advisor and mentor for our project work. We also express our sincere gratitude to our co-guide, Prof. S.C. Mishra, HOD, Metallurgical and Materials Engineering for providing valuable departmental facilities and also guide us.

We are very thankful to Prof. I. Samajdar of IIT Bombay for his benevolent permission for using the EBSD analysis facility at IIT Bombay for conducting experiments and also to Prof. P. Pant of IIT Bombay for her help in nanoindentation experiment. We are also thankful to Prof. S. Suwas of IISc Bangalore for his help in rolling operation experimentation.

Finally, we are indebted to all those who have directly or indirectly supported us to take this project work successful.

Place: Rourkela

Brahmananda Hota
111MM0363

Pravat Kumar Sahoo
111MM0379

Nabodit Patra
111MM0576

ABSTRACT

Pure magnesium was subjected to cold rolling and annealing at 200^{o}C for 30 min of soaking time. The annealed sample was then characterized by nanoindentation and electron backscattered diffraction (EBSD) to estimate the orientation dependent hardness in pure magnesium. It was observed that the grains of basal orientations had higher hardness value in pure magnesium. An increased deviation from exact basal orientation had decreasing trend on hardness value. Correlation of orientation and mechanical properties (particularly hardness) was investigated in the present study.

KEYWORDS: Magnesium, Orientation, EBSD, Nanoindentation, Hardness.

Content

List of Figures

CHAPTER

1

Introduction

1.1 Introduction

Magnesium and its alloys have a great potential for lightweight structural applications due to their high specific strength, low density, good weldability, corrosion resistance, good castability, turned/milled at high speed, and specific stiffness [1]. Magnesium and its alloys are most widely used in many structural applications such as automobile, aircraft, military, jet engine etc [2,3]. It has also been used as a potential material for laptop/computer body, flash photography, etc. Magnesium is the most useful metal in structural application after steel, aluminium and titanium. However, their poor formability at room temperature is an important limitation of magnesium for its usage as a structural material for it's HCP structure [4,5]. Various workers have tried to improve its room temperature formability through grain refinement, modifying its texture through different thermo-mechanical processing etc [6]. Mechanical properties of magnesium and its alloys very strongly depend on it's orientation due to its inherent anisotropy property of the hexagonal crystal structure. Mechanical forming such as rolling, forging and extrusion specially tends to the development of a strong orientation/texture with the basal plane perpendicular to the deformation direction [7]. The objective of the present study is to determine the hardness of different orientations in pure magnesium through nanoindentation. In this way an attempt is to be made to correlate the mechanical property of pure magnesium with its texture/orientation. Because magnesium and its alloys strongly depends on texture due the inherent anisotropy of closed packed HCP crystal structure which having less slip plane[8,9].

Nanoindentation is the most developed technique for extracting hardness and elastic modulus of the materials at nanometer distances [10]. This technique developed by Oliver and Pharr measuring mechanical property of the soft materials [10-13]. Nanoindentation hardness measurement is very small scale and is highly designed to provide good, accurate and repeatable results. It gives result very quickly as compared to other hardness tester/instrument [14]. As compared to other hardness tester it gives typical advantages such as, a precise value of the hardness of each phase of materials. Berkovich indenter is commonly used due to it's various advantages. It has been also investigated that nanoindentation technique with a hard film on a softer substrate by the finite element method, and found that the increase in both the yield strength ratio and the indenter tip radius increases the ratio of critical thickness to penetration depth. During testing indenter is driven in to the material and both plastic and elastic deformation occur suddenly. A Load – Displacement curve is sketched as per indentation data. According to Oliver and Pharr hardness and elastic modulus are derived form from the slope of the upper portion of unloading curve and instrumented hardness is measured by applied load divided by contacted area[15].In this experiment achievement of hardness from individual grains via EBSD technique. Such technique is very much useful for easy to identifying the crystallographic orientation of individual grains of polycrystalline materials [16]. In this project EBSD is directly used to investigate the mechanical property influence of crystallographic orientation on pure magnesium.

1.2 Objective of Work

The objectives which are to be achieved in this project are:

* Identification of crystallography orientation of individual grains from annealed electro polished pure Magnesium by EBSD.
* To determine the hardness of annealed pure Magnesium by nanoindentation.
* Correlation of orientation dependent hardness in annealed pure Magnesium.

1.3 Framework of the thesis

This thesis is divided into five chapters.

Chapter-1: Concerns about the introduction of the project work.

Chapter-2: Gives theoretical overview of Nanoindentation of materials, its measurement technique, different types of indenter with example and EBSD representing texture developed in materials.

Chapter-3: Represents the details of Magnesium and sample preparation followed by characterization techniques used in the present investigation.

Chapter-4: Basically tells the results that are obtained by texture measurements, hardness and discussion of the experimental results obtained.

Chapter-5: Description of comprehensive summary and the scopes for further work

CHAPTER

2

Literature Review

2.1 Nanoindentation theory and measurement

2.2 Types of indenters

2.3 Orientation and its Importance

2.3.1 Electron backscattered diffraction (EBSD)

2.4 Brief Literature Review on Nanoindentation and Material Property

2.0 Literature Review

2.1 Nanoindentation theory and measurement

Nanoindentation is a technique, which is used to measure the mechanical property the materials particularly for thin films but at a very small range (Micron to Nano). The principal major instruments in nanoindentation operation are the material sample, sensors and actuators. These are generally used for experiment and to measure the applied mechanical load and the displacement of indenter, and the edge of indenter for indentation. Further the important component is normally shaped as diamond, fiction to a sharp, similar shape such as the three-sided Berkovich pyramid. The depth of penetration is in **nm** range whereas applied load is in **µN** range. In such indentation technique, the extent of leftover response is just a few micron ranges which makes it is largely troublesome to get an exact measurement utilizing visual-optical procedures. Such type of analysis is regularly observed using instrumented machines with parameters such as indenter load (P) and displacement (h) can be recorded with progressively and simultaneously during loading and unloading condition. After that the response results are figure out to result-out the strength/hardness (H)and the Young's/elastic modulus (E).

Load – Displacement curve: In a normal technique, applied force and penetration depth are records as activated load set up from zero to maximum and vice versa from it's reverse condition. The shape of loading curve is a combined effect of elastic cum plastic and unloading curve is only effect of elastic effect. The holding is depends on type of material and temperature. At the point when burden is evacuated from the indenter and the materials effort to recover of that unique geometry, yet it kept from doing so as a result of plastic twisting. Be that as it may, there is some level of recuperation because of the unwinding of versatile strains inside the material. An investigation of the introductory segment of this versatile emptying reaction provide an evaluation of the soft modulus of examined material. The type of the consistence bends for the almost always widely recognized sorts of such indenter are much alike which is demonstrated in figure 2.1.

Nanoindentation technique developed described an useful method to manipulate the experimental mechanical responses. In indentation data analysis mainly it's hardness and Young's modulus are determined by the popular method known as Oliver and Pharr method, which is considered as important popular standard method [7]. This instrumented indentation technique (IIT) has been widely selected and used in the characterization of small-scale mechanical behavior. Oliver-Pharr method considered as one of the standard method for depth indentation technique. No other method has invented for such technique. This method generalized from figure 2.1.

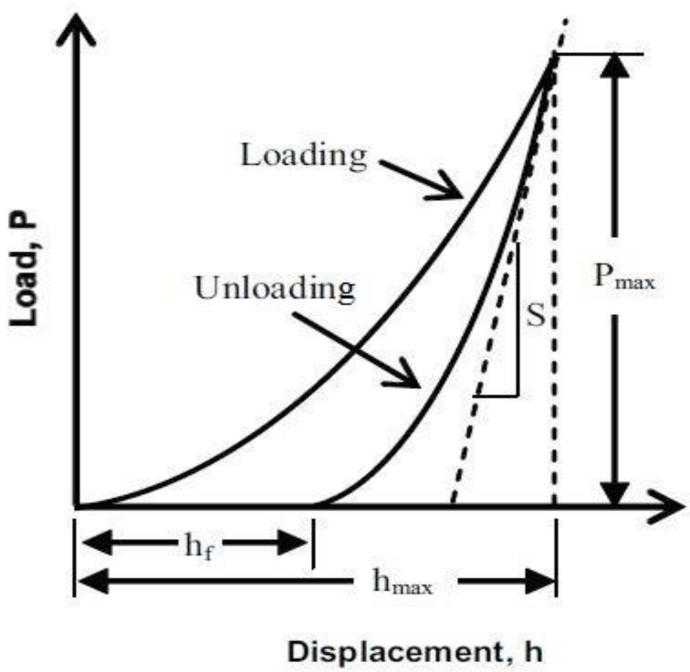


Fig. 2.1 A schematic representation of load displacement curve (Stiffness S, maximum load L_{max}, maximum depth h_{max}, final depth h_f and maximum load Pmax)[17]

The assumptions of the indentation method is observed to solve as follows [18,19]: From the Oliver-Pharr method, determination of hardness is as follows

$$H = \frac{P_{max}}{A} \tag{1}$$

Where H is Hardness of material, P_{max} is Maximum applied load and A is Area of contact

Determination of Young's modulus by the mathematical expression

$$E^* = \frac{(\sqrt{\pi}\ S)}{(2\beta\ \sqrt{A})} \tag{2}$$

Where E^* is Young's modulus, S is contact stiffness, A is area of contact and β is correction coefficient (1.034 for Berkovich)

And $$\frac{1}{E^*} = \frac{1-\nu}{E} + \frac{1-\nu_i}{E_i} \tag{3}$$

Where ν and ν_i be the Poisson's ratios of the samples and the indenter respectively, and E and Ei be the corresponding elastic modulus. For diamond indenter, E_i =1141 GPa and ν_i =0.07 [7]

But contact stiffness is calculated on differentiation of unloading part of Load – Displacement curve. Contact stiffness, $S = \left(\frac{dP}{dh}\right)_{P=P_{max}}$ (4)

Now, Area of contact, $A_c = C_0\ h_c^2 + C_1 h_c + C_2\ h_c^{1/2} + C_3\ h_c^{1/4} + C_4\ h_c^{1/8}$..... (5)

Here Ac is area of contact, C_0 is tip supplier and C_1, C_2, C_3, C_4 …are tip coefficient, and

$$h_c = h - \varepsilon\frac{P}{S} \tag{6}$$

Where h_c is depth during load, h is displacement of the tip, Ɛ is a constant which depends upon the types of indenter (0 .75 in diamond), P is applied load and S is contact stiffness

Again $P = B\ (h_{max} - h_f)^m$ (7)

Where P is applied load, B and m are experimental coefficient, h_{max} is maximum depth of penetration, h_f is final depth of penetration.

Now put the value of 'P' in equation (4), we get

Contact stiffness, $S = \left(\frac{dP}{dh}\right)_{P\,=\,P_{max}} = Bm\,(h_{max} - h_f)^{m-1}$ (8)

2.2 Type of indenters

There are various geometries available for indenter process. According to tip geometry indenters are devided into two types named as sharp or blunt. Indenter are mainly differ by its tip and geometry shape. Depth of testing indentation can be performed by micron scale to nanoscale. Five main different categories of indenter tips are now available for indentation instrument testing (IIT). It is very important to choose desired tip as per suitable application. Which gives you desired accurate result with in few second. Its tip radius can be evaluated by powerful technique like SEM or AFM. Diamond indenter is the most popularly used for indentation due to its desired properties such as hardness, chemical inertness and thermal conductivity.

• Berkovich

The Berkovich indenter is commonly used for most indentation instrument testing (IIT) to estimate mechanical property in nanoscale. It is a three sided pyramid shape with face angle 65.27^0 which having self-similar geometry. Such type of geometry is usually favored which is a four-sided pyramid popularly named as Vickers indenter. It is cannot be break easily and very quickly prepared. It generate plasticity at very small range loads which provides a meaningful measurement of hardness. It's indenter tip has a high included angle of 142.3° which reduces the consequence of friction.

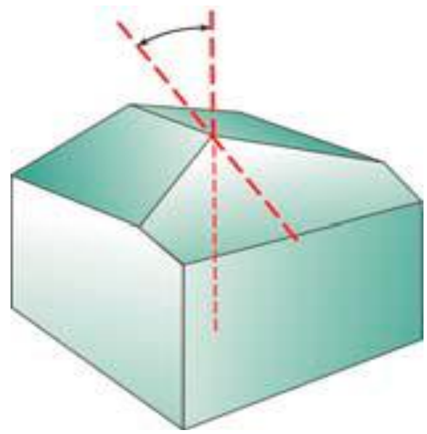


Fig.2.2 Berkovich indenter tip [20]

Applications -

- Thin films
- Bulk materials
- Polymers
- Micro-electromechanical Systems (MEMS)
- Scratch Testing
- Wear Testing
- In-situ process Imaging

• Vickers

A Vickers nanoindenter is used for instrumented indentation testing (IIT) to measure mechanical properties of material's on nanoscale. The geometry of it's tip is a square pyramidal shape. The geometry of vicker indenter tip having faces angle136°.

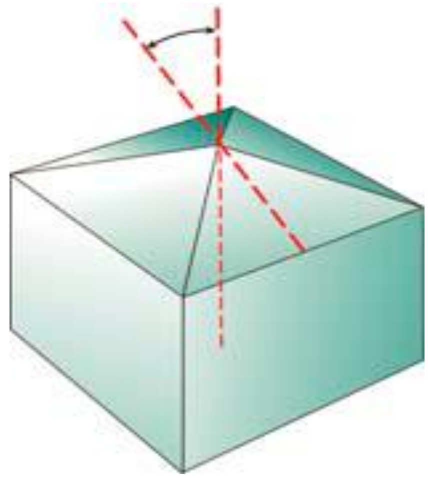


Fig.2.3 Vicker indenter tip [20]

Applications -

- Bulk Materials
- Films and Foils
- Scratch Testing
- Wear Testing

• Cube Corner

The Cube Corner indenter is built with three-sided pyramidal shape with which jointly perpendicular faces are arranged in a geometry like joint of cube corner. The centerline-to-face angle is 34.3°. The sharpness tip of the cube corner generates much higher stresses and strains on the applied contact area. This is much more useful for testing in brittle material for very small, well-defined cracks around hardness impressions. Fracture toughness can be measure by using this crack at a micro scales.

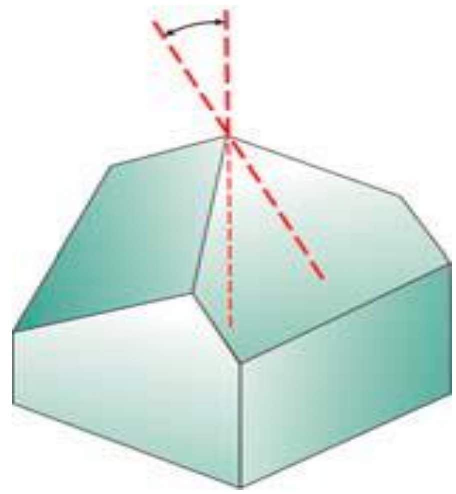


Fig.2.4 Cube corner indenter tip [20]

Applications -

- Thin Films
- Micro-electromechanical Systems (MEMS)
- Scratch Testing
- Wear Testing
- Fracture Toughness
- In-situ Imaging

• Cone

The conical indenter drawing a sharp and self-symmetric geometry but cylindrical symmetry makes it attractive from a modeling point of view. Other reason for more attractive because of the complications associated with the stress concentrations at the sharp edges of the indenter are absent. Nonetheless a little bit IIT testing has been conducted by this. The reason behind the limitation is that very complex to fabricate with sharp tip. The complications does not occur at large scale where much should be tested by applying conical in IIT testing. Such type of indenter is very rarely used for IIT, as its disadvantage is described earlier. So its application is limited on such field.

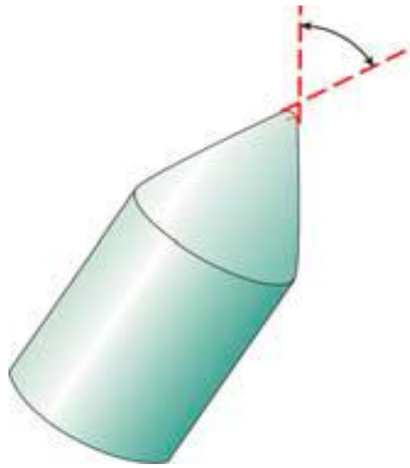


Fig.2.5 Cone indenter tip [20]

Applications -

- Scratch Testing
- Wear Testing
- Micro-electromechanical Systems
- In-situ Imaging

• Sphere

The Spherical indendenter is also increasing popularity after diamond indenter. Such type of indenter results out a very smooth transformation from elastic to plastic contact. This simulate damage of contact during in in-service conditions so this is especially suitable for soft materials measurement. Indentation instrumented technique with spheres has most successfully occupied with larger-diameter. But in the micro range applicatio of spherical indenters is difficult in accessing high-quality spheres which is fabrication from rigid and hard materials. Due to the limitation of this reason Berkovich indenters are highly preferred.

Fig.2.6: Sphere indenter tip [20]

Application -

- Micro-electromechanical Systems (MEMS)

2.3 Orientation and its Importance

Orientation or crystal orientation means the relative positioning of atomic plane in a crystal to a fixed reference frame. Determination of topography of crystallite orientations is an important technique for study of polycrystalline materials by powerful technique. Kikuchi patterns is used for orientation determination for a long time [21]. Fully automated generated of orientation detacted by electron back scattered diffraction (EBSD) is a well-traditional technique. Pole figure and inverse pole figure curve analysis are the most commonly useful techniques for orientation. From these two figures hardness of separate orientation in particular axis very easily analyzed. From research it has been found that that crystallography orientation is important in surface process such as corrosion, dissolution or oxidation. The effect of crystallography orientation is related to binding energy of the surface atom [22].

In a polycrystalline material crystallography orientation spreading is highly unlikely to be randomly distributed called texture. Texture is observed in almost all engineering materials which influence the mechanical property of materials. The texture itself is generated by the forming process (wire drawing or sheet rolling) is called deformation texture. Due to the tendency of the grains in a polycrystalline aggregate to rotate during the plastic deformation. Every individual grains undergo slip formation and rotates in a complex way that can be observed by the appoint forces and by the slip system and rotation of adjoining grains and the result should be a preferred nonrandom orientation. When any cold-worked material, undergoes of a deformation texture, which recrystallized by annealing, the new grain structure has a preferred orientation which is different from that of the cold-worked material. This is called an annealing texture/ recrystallization texture [23]. It is due to the nucleation and/or growth of the new grains in that matrix.

The importance of preferred orientation lies in the effect, often very marked, which it depends on the overall, macroscopic properties of materials. For indentation testing preferred orientation is beneficial for determining the mechanical property of materials.

2.3.1 Electron backscattered diffraction (EBSD)

Electron backscatter diffraction also known as backscatter Kikuchi diffraction (BKD) is a very powerful Scanning Electron Microscope (SEM) based microstructural technique to characterizing the crystallographic orientation. In other word EBSD is an unique technique that it provides a link between microstructure and crystallography orientation. However, even with the speed of modern EBSD technique, the collection times required to obtain the orientation data precludes the use of orientation imaging microscopy (OIM) mapping as an imaging tool in the conventional sense. It is promoted to focus the crystallographic revels of various types materials, which might be utilized to depict surface or favored introduction of any crystalline or polycrystalline material. EBSD can used to index and distinguish all the seven crystal frameworks. So it takes place to connected with Crystal orientation, Grain morphology, local heterogeneity, global and local texture, micro strain mapping, physicochemical ID.

EBSD scan is carried out utilizing a Scanning Electron Microscope (SEM) outfitted with an EBSD indicator which is linked with a phosphor screen, minimal lens and low light CCD Polaroid chip. The current cameras employed as EBSD detectors are CCD (charge-coupled device) cameras. The single advantages of CCD cameras is the ability to combine the charges from adjacent pixels into a single readout signal. The CCD chip with a local determination of 640×480 pixels is utilized for quick estimations, while for slower, and more delicate estimations the CCD chip determination can go up to 1600×1200 pixels. Thus CCD camera resolution also can be effect the integration time for composes diffraction pattern. For composition and introduction estimations, the pictures are binned so as to reduce their size and computational times. The exchange and elucidation are up to just about 1000 pictures/s is conceivable, if the diffraction sign is sufficient.

EBSD is very sensitive to crystalline perfection so, a nicely prepared polished sample is a prerequisite for achieving a good diffraction pattern. For good EBSD estimation a cleaned crystalline sample is put in the SEM chamber with tilted angle 70° from the horizontal axis to generate patterns of sufficient intensity. Also for this an appropriate working distance, usually in the range 5 to 30 mm. The phosphor screen is spotted inside the sample assembly of the SEM at an edge of more or less 90° to the shaft piece and is coupled to a smaller lens which centers the picture from the phosphor screen to the CCD Polaroid. In such type of design a percentage of the electrons entering to the sample and backscatter may be escape. As these electrons leave from the sample, they may retreat at the Bragg's condition ($2d\ sin\Theta=n\lambda$) identified with the dispersing of the occasional nuclear cross section planes of the crystalline structure and diffract.

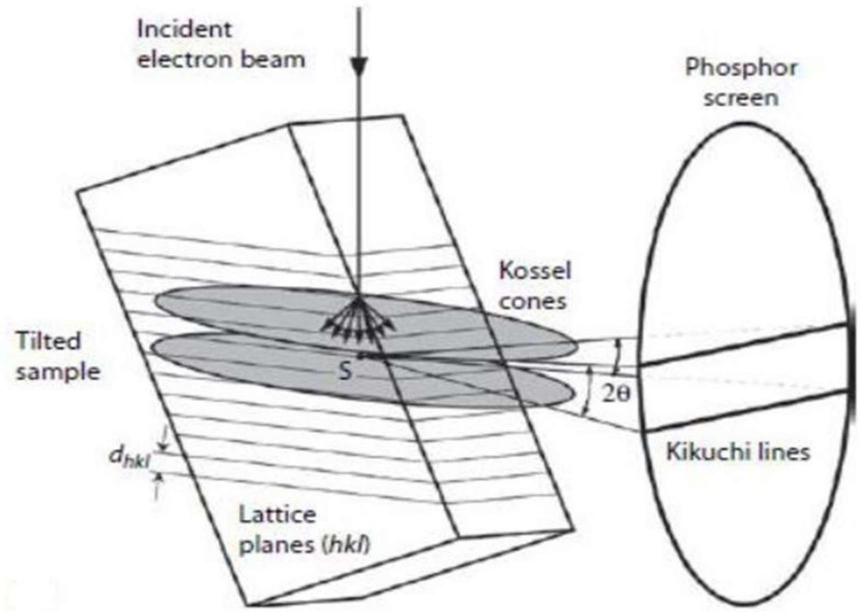


Fig.2.7 Origin of Kikuchi lines from the EBSD [24]

An electron backscatter diffraction patterns (EBSP) is formed when numerous distinctive planes diffract diverse electrons to structure Kikuchi groups which relate to each of the grid diffracting planes. Each diffraction patterns are collections set of Kikuchi bands which characterizes the sample crystal structure and it's orientation. Each Kikuchi Centre lines band are correlated to the junction with the phosphor screen of the diffracting plane which is responsible for the band formation. So, each Miller indices of the diffracting crystal plane can be indexed by the Miller indices .[25]

While the geometric illustration identified with the kinematic result (by utilizing the Bragg's law) is both influential and helpful for introduction and its composition investigation. Cross section depicted by the geometry and disregards various physical methodologies, which included inside the diffracting materials. To sufficiently characterize better characteristics inside the EBSD, which is one requisite utilize a various pillar dynamical model.

2.4 Brief Literature Review on Nanoindentation and Material Property:

Nanoindentation is popularly useful instrument for thin film material's property. This instrument gives hardness of each grains. Now-a-days nanomaterial is very important for both engineering as well as research purpose.

According to Yeol Choi *et al*[26] studied about the nanoindentation to characterized the strength of micro phase in Ultra-fined-grained steel. Equiaxed and polygonal shape of fine ferrite grain size 1-2 μm had higher hardness and elastic modulus than coarse grain. This is due to presence of Martensite phase and low dislocation density in API X65 steel.

C.-L. Chen *et al*[27] conducted an experiment that nanoindentation was used to carried out the mechanical characteristics of separate phases in multi-component of Al–Si alloys. As a result it was found that both hardness and reduced elastic modulus was increased as the Ni content of the Al–Cu–Ni phases also increases. This can be correspond with the formation temperature of intermetallic phases as a result with formation of high heat, which having a strong stable binding between atoms, and due to presence of their high elastic modulus.

Nanoindentation procedure of Cu thin films was investigated by S.H. Hong *et al*[28] to characterized the elastic moduls present in both perpendicular and parallel directions from the texture. Texture was analyzed by micro-cantilever beam bending test. The theoretical elastic modulus of perpendicular direction could be predicted by Voigt's model, while in the parallel direction could be predicted by Hill's model. Here it was observed that the elastic modulus is based on the texture analysis.

C. Fizanne-Michel *et al*[29]sketched that nanoindentation is point out to determine the relationship between grain orientations and hardnesses or elastic moduli by drawing inverse pole figures from commercially pure titanium. The result indicated that the hardness varies significantly with orientation, and the elastic modulus appears less sensitive than hardness to grain orientation. Hardnesses of grains present at basal plane possess higher values than at prismatic plane.

According to Bo Yang and Horst Vehoff [30]nanoindentation of nano nickel was used to study the hardness of each separate grains. The hardness with the dislocation density in the range whereas the indent size was smaller than the grain size, when a critical load was reached in a single grain, result for dislocation emission in adjacent grains could be clear from later pop-ins in the load–displacement curve. So the hardness not only depends on the grain size as well as also depends upon the ratio of grain size to the indent size.

Form these it is clear that nanoindentation is used to determine the mechanical property from individual grains of the materials. The hardness varies with phase changes of the materials. A fined grained material has stronger mechanical property than a coarse grained. SEM/AFM is commonly used for texture analysis, which is a more powerful technique. EBSD is a common technique for microstructure evaluation on crystallographic orientation.

CHAPTER

3

Experimental procedures

3.1 Material and Sample Preparation

3.2 Electron Backscattered Diffraction (EBSD)

3.3 Nanoindentation

3.0 Experimental procedures

3.1. Material and Sample Preparation

Pure magnesium was subjected to cold rolling of 90% reduction in thickness in a laboratory rolling mill at IISc Bangalore. The rolled plates are then subsequently annealed at 200^{o}C for 30 min followed by air cooling. Then the samples are electro-polished for different characterizations. Electro polishing process was performed by using an electrolyte containing a mixture of ethanol to ortho-phosphoric acid by 3:5 ratio (by the volume) at 0^{o}C. Initially the electro-polishing is carried out at 3 V for 30 sec and subsequently at 1.5 V for 2 minutes.

3.2. Electron Backscattered Diffraction (EBSD)

EBSD, on the ND plane of the magnesium samples, was performed on a FEI-Quanta 200-HV SEM (Scanning Electron Microscope) at IIT Bombay. Data acquisition and analyses ware carried out using the TSL-OIM version 6.0 software. Both beam and video conditions were kept identical distance between the scans and a step size of 0.2µm was used.

3.3. Nanoindentation

Nanoindentation is performe using a nano-mechanical testing instrument, Hysitron Triboindenter (TI 900) at IIT Bombay. A Berkovich diamond indenter was used for indentation. Hardness of different grains was measured using a load of µN range.

CHAPTER

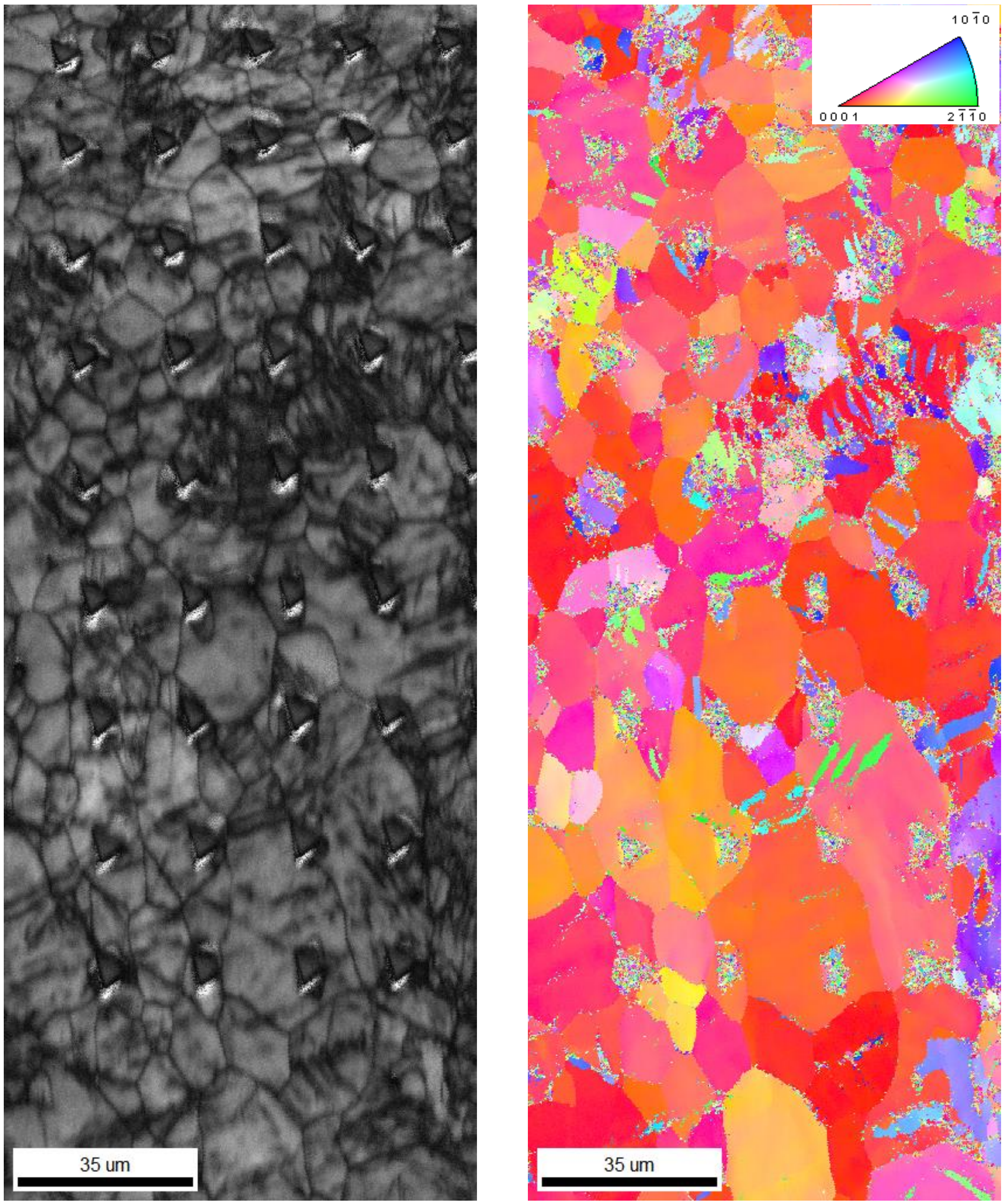
4

Results and Discussion

4.0 Results and Discussion

Figure 4.1 shows the EBSD microstructures of annealed magnesium after nanoindentation. The microstructures are represented by image quality map (Figure 4.1(a)) and inverse pole figure map (Figure 4.1(b)). It may be noted that this has been achieved by making nanoindentation on electropolished magnesium and then analyzing by EBSD on the same area where nanoindentation was performed. From the inverse pole figure maps the orientation of the grains can be identified. In this way the hardness of different grains or orientations in pure magnesium can be measured.

Figure 4.2 shows the inverse pole figure indicating the orientation of grains where nanoindentation was performed. The corresponding hardness of different grains/orientations of pure magnesium is shown in Figure 4.3. This clearly shows a higher hardness value for basal grains and an increased in deviation from basal orientation decreased the hardness value in the samples. It may be expected that when the indenter along the c-axis of a crystal i.e. when the grain/crystal is oriented along basal orientation, it is difficult to slip as the major slip system at room temperature deformation of pure magnesium is basal slip only. Hence, the hardness was found to be higher in basal grains/orientations in pure magnesium.



(a) **(b)**

Figure 4.1 Micrographs showing nanoindentation on annealed magnesium: (a) Image quality map and (b) Inverse pole figure map.

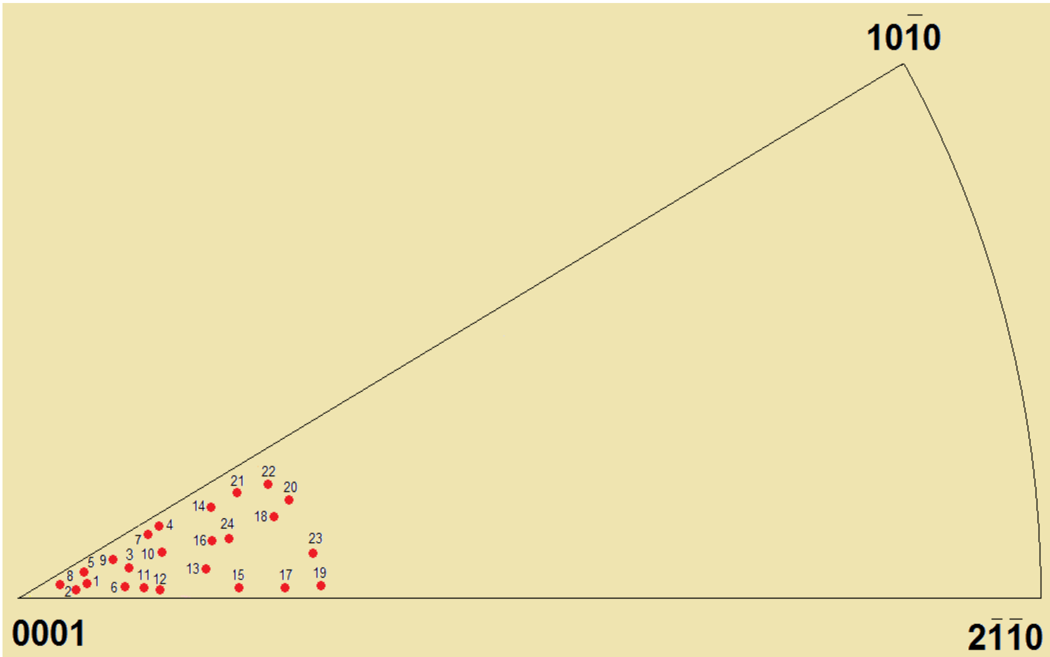


Figure 4.2. **Discrete inverse pole figure representing the grains/orientations where nanoindentation was carried out.**

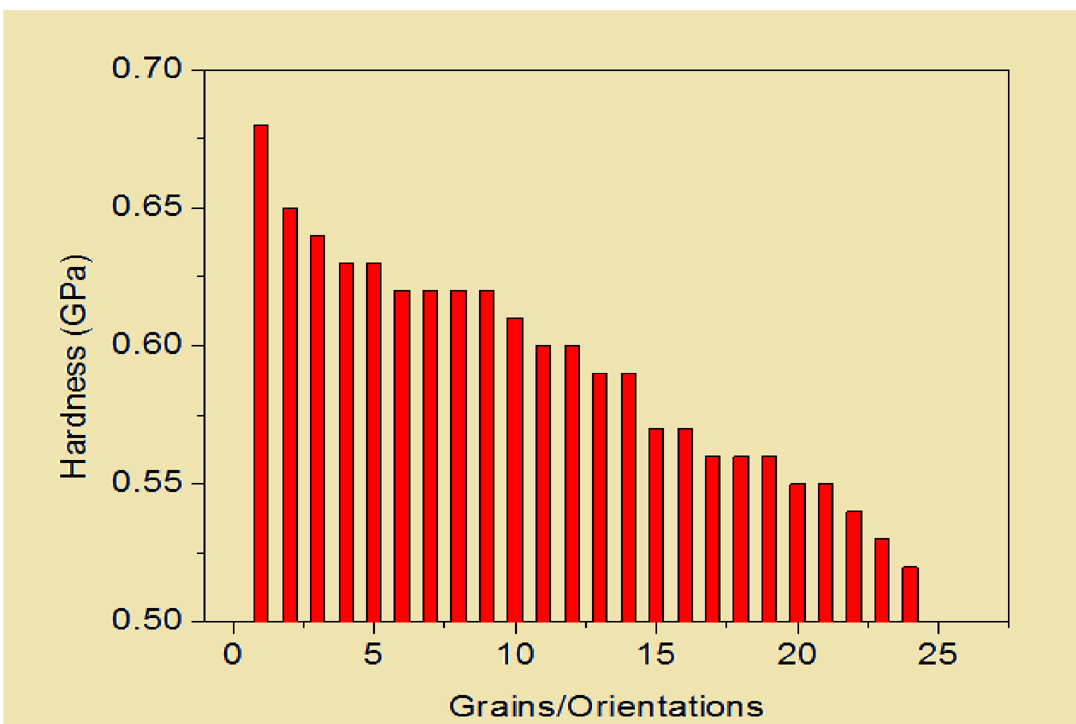


Figure 4.3. Corresponding hardness of different grains/orientations shown in figure 4.2.

CHAPTER

5

Summary and Scopes for future work

5.1 Summary

5.2 Scopes for future work

5.1 Summary:

The following conclusions may be made from the present study:

- The basal/near basal orientations were the hardest orientations in annealed pure Magnesium.
- Non-basal/away from basal orientations were the less hard orientations in annealed pure Magnesium.
- An increased deviation from exact basal orientation had decreasing trend in hardness values of pure magnesium.

5.2 Scopes for future work:

In this study we found that texture/orientation had strongly influence on mechanical properties of magnesium. However, the present study was focused on hardness value only. The other mechanical properties such as tensile strength, fatigue strength and impact strength etc. may be investigated to conclude the orientation dependent mechanical properties of pure magnesium or other magnesium alloys.

6.0 References

[1] Mordike B.L., Ebert T., "Magnesium Properties — applications — potential", *Materials Science and Engineering A*, vol.302 (2001), pp.37–45

[2] Czerwinski F., "Controlling the ignition and flammability of magnesium for aerospace applications", *Corrosion Science*, vol.86 (2014), pp.1–16

[3] Luo A.A., "Magnesium casting technology for structural applications", *Journal of Magnesium and Alloys*, vol. 1 (2013), pp. 2–22

[4] Lee B.D., Beak U.H., Lee K.W., Han G.S., Han W.H., "Protective Properties of SF6 under Various Carrier Gases for the Protection of Molten Mg", *Materials Transactions*, vol.54No. 1 (2013), pp.66-73

[5] Chen R., Liang S., Wu D., Han E., "Consideration of Castability and Formability for New Magnesium Alloys", *Scientific research*, vol. 2 (2012), pp. 8-17

[6] Somekawa H., Mukai T., "Effect of grain refinement on fracture toughness in extruded pure magnesium", *Scripta Materialia*, vol.53 (2005), pp.1059–1064

[7] Biswas S., Suwas S., Sikand R., Gupta A.K., "Analysis of texture evolution in pure magnesium and the magnesium alloy AM30 during rod and tube extrusion", *Materials Science and Engineering A*, vol. 528 (2011), pp. 3722–3729

[8]. Hutchinson W.B., Barnett M.R., "Effective values of critical resolved shear stress for slip in polycrystalline magnesium and other hcp metals", *Scripta Materialia*, Vol. 63 (2010), pp.737–740

[9]. Staroselsky A., Anand L., "A constitutive model for hcp materials deforming by slip and twinning: application to magnesium alloy AZ31B", *International Journal of Plasticity*, Vol. 19 (2003), pp.1843–1864

[10] Jian S.R., Huang C.Y., Ke W.C., “Nanoindentation responses of InN thin films”, *Journal of Alloys and Compounds*, vol.609 (2014), pp.125–128

[11] Yan W., Pun C.L., Simon G.P., “Conditions of applying Oliver–Pharr method to the nanoindentation of particles in composites”, *Composites Science and Technology*, vol. 72 (2012), pp. 1147–1152

[12] Ahn J.H., Kwon D., “Micromechanical estimation of composite hardness using nanoindentation technique for thin-film coated system”, *Materials Science and Engineering A*, vol.285 (2000), pp.172–179

[13] Zhoua L., Yao Y., “Single crystal bulk material micro/nano indentation hardness testing by nanoindentation instrument and AFM”, *Materials Science and Engineering: A*, Vol. 460–461, (2007), pp. 95–100

[14] Yan W. , Pun C.L., Wu Z., Simon G.P., “Some issues on nanoindentation method to measure the elastic modulus of particles in composites”, *Composites Part B: Engineering*, Vol. 42 (2011), pp.2093–2097

[15] Li X., Bhushan B., “A review of nanoindentation continuous stiffness measurement technique and its applications”, *Materials Characterization*, vol. 48 (2002), pp.11–36

[16] Moreau A., “Evaluation of macrozone dimensions by ultrasound and EBSD techniques”, *Materials Characterization*, vol. 75, (2013), pp.115–128

[17] Nayyeri G., Poole W.J., Sinclair C.W., “Investigation of the local mechanical properties in pure Mg using nanoindentation” Mg2012: 9th International Conference on Magnesium Alloys and their Applications.

[18] Zhou X Y, Jing Z D, Wang H R, Zhu Q, "A Method to Extract the Intrinsic Mechanical Properties of Soft Metallic Thin Films Based on Nanoindentation Continuous Stiffness Measurement Technique", *Journal of Physics*, vol.48 (2006), pp.1096–1101

[19] Lucca D.A., Herrmann K., Klopfstein M.J., "Nanoindentation: Measuring methods and applications", *Manufacturing Technology*, vol.59 (2010), pp.803–819

[20] Dash K., "Processing and characterization of Cu-Al_2O_3 and Al-Al_2O_3 composie: An evolution for Micro and Nano particulate Reinforcement", Ph.D Thesis NIT Rourkela (2014)

[21] Fundenberger J J, Morawiec A, Bouzy E, Lecomte J S, "Polycrystal orientation maps from TEM", *Ultramicroscopy*, vol. 96 (2003), pp. 127–137

[22] Liua M., Qiua D., Zhaoa C. M., Songa G., Atrensa A.,"The effect of crystallographic orientation on the active corrosion of pure magnesium", *Scripta Materialia*, vol.58 (2008), pp. 421-424

[23] Wright S.I. , Nowella M.M., Kloeb R., Camusc P., Ramptonc T., "Electron imaging with an EBSD detector", *Ultramicroscopy*, Vol.148 (2015), pp.132–145

[24] Engler O. and Randle V., "Introduction to texture analysis", Florida, CRC Press, 2000

[25] Deal A., Hooghan T., Eades A., "Energy-filtered electron backscatter diffraction", *Ultramicroscopy*, vol. 108 (2008), pp. 116–125

[26] Selvarajou B., Shin J.H., Ha T.K., Choi I., Joshi S.P., Hand H.N., "Orientation-dependent indentation response of magnesium single crystal: Modeling and experiment", *Acta Materialia*, vol.81 (2014), pp. 358-376

[27] Chen C.L., Richter A., Thomson R.C., "Mechanical properties of intermetallic phases in multi-component Al–Si alloys using nanoindentation", *Intermetallics*, vol. 17 (2009), pp.634–641

[28] Hong S.H., Kim K.S., Kim Y.-M., Hahn J.-H., Lee C.-S., Park J.-H., “Characterization of elastic moduli of Cu thin films using nanoindentation technique”, *Composites Science and Technology*, vol. 65 (2005), pp.1401–1408

[29] Fizanne-Miche C., Cornen M., Castany P., Péron I., Gloriant T., “Determination of hardness and elastic modulus inverse pole figures of a polycrystalline commercially pure titanium by coupling nanoindentation and EBSD techniques”, *Materials Science & Engineering A*,vol.613 (2014),pp.159–162

[30] Yang B., Vehoff H., “Grain size effects on the mechanical properties of nanonickel examined by nanoindentation”, *Materials Science and Engineering A*, vol.400–401 (2005), pp.467–470