# Novel Method by Vickers Hardness to Determine Mechanical & Microstructural Parameters Using GNDs & SSDs

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## Abstract:

Aluminum alloys are gaining more ground as first choice materials, especially in the transportation industry where a high strength to weight ratio is of premium importance. 3004 aluminum alloy is one of the most used non-heat treatable alloys which are employed in many industries (aeronautic, aerospace, blades, discs, rings, airframes…etc.), due to its attractive mechanical properties. In this work, the mechanical parameters of 3004 aluminum alloy i.e hardness coefficients (the total hardness, hardness of GNDs and hardness of SSDs) in addition to the microstructural parameters (mean crystallite size, micro strain and dislocation density) were determined from Vickers hardness measurement by using novel methods. The flow stress and stored energy were also highlighted.

## Keywords:

Hardness Test, 3004 Aluminum Alloy, Dislocation Density, Defect Density, Total Hardness, GNDs, SSDs, Estimated Crystallite Size, Micro Strain, Stored Energy

## 1. Introduction

Currently, the needing for aluminum alloys that offer good performance at different deformations is spreading, driven by the automotive and aerospace manufacturing requiring higher toughness, strength, lower cost and lighter structural and microstructural components. Aluminum alloys can generally be classified as wrought alloys and casting alloys. 3004 aluminum alloy includes the non-heat treatable wrought series [1,2]. A dislocation is one kind of defects present in crystals, or irregularity, within structure of crystals. The presence of dislocations strongly affects many of the properties and characteristics of materials. The motion of dislocations can be produced if the atoms presented in surrounding planes break their bonds and rebound with the atoms at the terminating edge [1]. In many metals and alloys, particularly ductile materials, dislocations are the "carrier" of inelastic deformation, and the energy needed to move them is less than the energy needed to fracture the material. Dislocations give rise to the characteristic malleability of alloys and metals.

The density of dislocations and density of defects are key microstructural factors since it is widely related to mechanical properties of examined samples. The density of dislocations in tested alloy can be significantly altered during inelastic straining, thermal annealing or when they undergo plastic deformation such as cold pressing as in the case of tested 3004 Al alloy. In deformed alloys, the stored energy formed by dislocations is the driving force for important solid-state reactions like recovery and recrystallization [3]. Furthermore, the quantitative estimation of the density of dislocations in tested 3004 Al alloy is also very important in the development of theories of inelastic deformation [4]. There are many experimental and traditional techniques to evaluate mechanical and microstructural properties of materials including Vickers hardness testing, X-ray diffraction line profile analysis,…etc. The used experimental technique in this work is the Vickers hardness test. Hardness of metals and alloys rely widely on the indentation size and grain size via micro-indentation of non-deformed and deformed 3004 aluminum alloy [5-6]. The mechanical properties of investigated samples are measured by indentation tests which have widely been used as an effective and economical including Vickers hardness measurement [7]. It well known that indentation hardness relies on depth or the indentation size. This effect is referred to the indentation size effect (ISE) and is concerned to geometrically necessary dislocations (GNDs) which are present to accommodate the strain gradient [8]. It is appeared that in the Nix−Gao model the total density of dislocations represents the resultant coupling of GNDs and SSDs, both of which contribute a significant role in the hardening mechanism [9].

Hardness investigation can be known according to the applied forces and displacements obtained as micro-, or nano- scale. Hardness is the specific property of a material that makes it to resist plastic deformation, often by penetration. The Vickers hardness test consists of indenting the examined alloy with an indenter made from diamond, to make a right pyramid with a square base and an angle of 136 degrees between opposite faces subjected to a load of 1 to 100 kgf. The applied load used during Vickers hardness experiment is 4.9 N. The full load is normally applied for 10 to 15 seconds. The two diagonals of the indentation left in the surface of the material after removal of the load are measured using a microscope and their average calculated. The area of the sloping surface of the indentation is calculated. The Vickers hardness is calculated by dividing the applied load by the square area of indentation [6]. In this work the hardness coefficients, density of dislocations, density of defects in addition to flow stress and stored energy were highlight.

## 2. Samples Preparation for Vickers Hardness Investigations

In this study, the needed samples of the given alloy were cut, as the first stage, with needed dimensions and sizes of (12 x 12 x 3) $mm^3$. 3004 aluminium alloy composition is cleared in Table 1. On the second stage, the samples surface was put under grinding with paper of order or size of 120, 220, 300, 600, 1000 and 1200 grit to make a smooth finish on flat surfaces. This stage makes us to reach a fine flat surface but soft enough. Polishing of samples which lies on the third stage was done to create a shiny and smooth surface by rubbing it or using a chemical action, leaving a surface with a significant specular reflection. The fourth stage is known as the annealing stage by which investigated specimens annealed at 450℃ in a furnace with no vacuum for eliminating injected stresses and then cleaned with needed solution and then dried. Finally the specimens were plastically deformed at states from 0.00% to 33% thickness reduction (at room temperature) to be ready for work. The mechanical characteristics of the prepared samples can be evaluated by Vickers hardness measurements as shown in Figure 1; it is the standard method for measuring the hardness of materials, especially those with extremely hard surfaces: the surface is subjected to a standard pressure for a standard length of time by means of a pyramid-shaped diamond. The diagonal of the resulting indention is measured under a microscope.

***Table 1.*** *3004 Al-alloy chemical composition.*

| Element | Si | Fe | Cu | Mn | Mg | Zn | others | Al |
|---|---|---|---|---|---|---|---|---|
| Wt % | 0.3 | 0.7 | 0.25 | 1.0-1.5 | 0.8-1.3 | 0.25 | 0.15 | reminder |

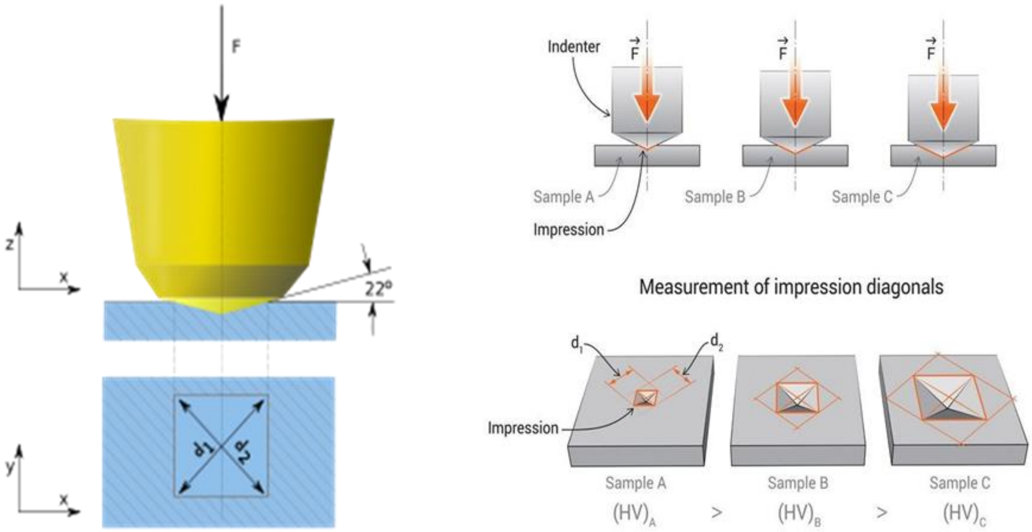


***Figure 1.*** *The tested samples under the Vickers hardness measurements.*

## 3. Results and Discussion

### *3.1. Theoretical Basics on Hardness Test*

The total hardness ($H_t$) can be presented as the summation of hardness due to internal friction ($H_{in}$), solid solutions ($H_{sl}$), dislocation density ($H_d$) and grain boundary ($H_g$) as follow:

$$H_t = H_{in} + H_{sl} + H_g + H_d \tag{1}$$

($H_g = 0$) can be neglected due to the size of indentation occur at the center of the grain and there is no interaction between dislocations and grain boundaries [10]. ($H_{in}$) is depth independent because we do not compare BCC by FCC metals.($H_{sl}$), is also a constant value such as to ($H_{in}$). Equation (1) could be rewritten for macro-hardness ( $H_0$) and micro-hardness ( $H_{mi}$),

$$H_0 = H_{in} + H_{sl} + H_{SSD} \tag{2}$$

$$H_{mi} = H_{in} + H_{sl} + H_{ISE} \tag{3}$$

Where, $H_{SSD}$ is the hardness resistance resulting only from SSDs and $H_{ISE}$ is the hardness resulting from the coupling of SSDs and GNDs. Nix and Gao [9] proposed $H_{SSD}$ and $H_{ISE}$ can be calculated using the following equations:

$$H_{GND} = H_{ISE} - H_{SSD} \tag{4}$$

$$H_{GND} = H_{mi} - H_0 \quad (5)$$

$$H_t = H_{ISE} = H_{SSD} + H_{GND} \quad (6)$$

### *3.2. Determination of the Hardness Coefficients*

The total hardness ($H_t$) is calculated by dividing the applied load by the square the area of indentation as follows:

$$H_t = HV = \frac{2F sin\frac{136}{2}}{d^2} = 1.854\ \frac{F}{d^2} \quad (7)$$

Where F is the applied load in kgf, d is the arithmetic mean of the two diagonals in mm and HV is the Vickers hardness. The variation of total hardness is depicted in Figure 2.

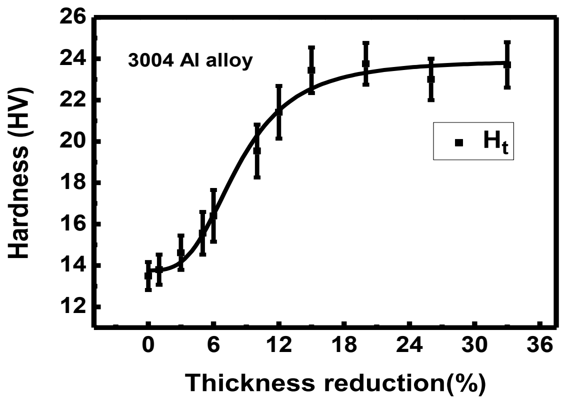


***Figure 2.*** *The total hardness ($H_t$) of 3004 Al-alloy as a function of thickness reduction.*

On the other, the hardness of GNDs can be calculated by subtracting the value of the hardness of SSDs (bulk hardness) from the values of the total hardness at each degree of thickness reduction according to equation (6). The variation of the hardness of GNDs is depicted in Figure 3.

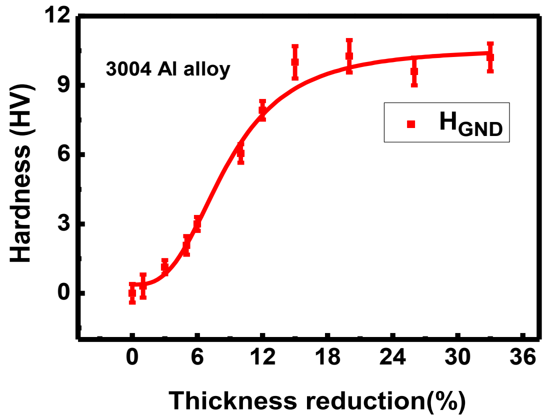


***Figure 3.*** *The hardness of GNDs, ($H_{GND}$), of 3004 Al-alloy as a function of thickness reduction.*

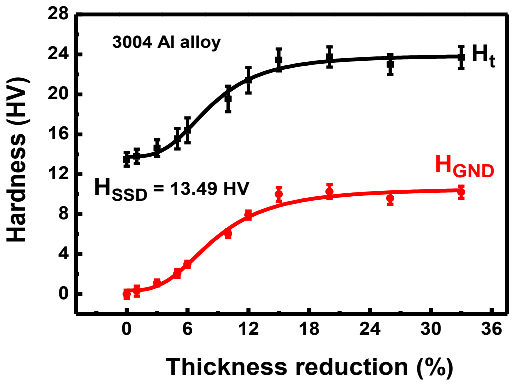


***Figure 4.*** *The hardness ($H_t$) and ($H_{GND}$) of 3004 Al-alloy as a function of thickness reduction.*

The hardness coefficients i.e the total hardness and the hardness of GNDs were changed exponentially as a function of thickness reduction as indicated in Figure 4. It is clear that, the total hardness ($H_t$) and the hardness of GNDs, ($H_{GND}$) increase slowly (exponentially) from 0.00 % to 6.00 % thickness reduction which corresponding to (13.49-16.4 HV) and (0.00-3.00 HV) respectively, then followed by fast increase in the range from 6.00% to 15.00% thickness reduction corresponding to (16.4-23.44HV) and (3.00-10.00HV) respectively, and finally, they become constant above this value. From Figure (4), the hardness of SSDs is $H_{SSD} = 13.49$ HV.

### *3.3. Evaluation of the Defect Properties*

When a metal or alloy is subjected to inelastic deformation (during hardness measurement) the dislocation density increases, leading to strain hardening. If no dislocations are found in the region of deformation, new dislocations must be nucleated for inelastic deformation to be produced. The density of dislocations is calculated from hardness measurements by the given formula [10]:

$$H_0 = 3\sqrt{3}\,\alpha\, G\, b\,\sqrt{\rho_{SSD_s}} \tag{8}$$

$$H_t = 3\sqrt{3}\,\alpha\, G\, b\,(\sqrt{\rho_{SSD_s}} + \sqrt{\rho_{GND_s}}\,) \tag{9}$$

$$\rho_t = (\sqrt{\rho_{SSD_s}} + \sqrt{\rho_{GND_s}}\,)^{\,2} \tag{10}$$

Where (α = 0.5) is constant depend on the shape of the grains and (G = 26 Gpa) is the bulk modulus of 3004 Al alloy. 1Gpa = 102 kgf or HV and (b = 0.286 nm) is burger vector of Al.

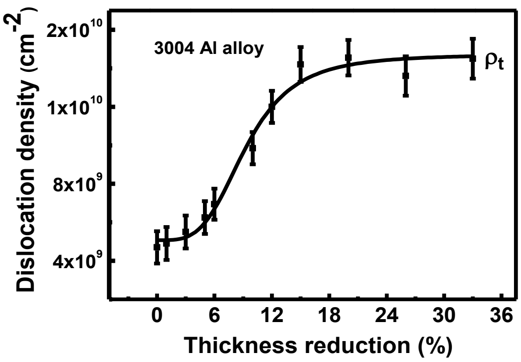


***Figure 5.*** *The total dislocation density ($\rho_t$) as a function of thickness reduction.*

By using the above equations, the total dislocation density ($\rho_t$) calculated by equation (9) exhibit exponential gross as indicated in Figure 5.

### *3.3.1. Derivation for the Density of GNDs*

The essential factors for indentation tests by conical/pyramidal indenter are (see Figure 6): the force applied to the indenter, F, the residual contact radius of indentation, $a_p$ , the hardness, $H = F/\pi a_p^2$, the permanent indentation depth, $h_p$, the total indentation depth, h , the plastic zone radius, $c_p$ , and the indenter geometry; i.e., the angle between the surface of the conical indenter and the plane of the surface $\theta$ . This angle is related to $h_p$ and $a_p$ by $\tan(\theta) = h_p/a_p$ (see Figure 6). The unloading process in the indentation experiment is essential for the proper specification of these geometric parameters. Thus, the residual values $h_p$ and $a_p$ should be used as measurable data in the hardness H calculations.

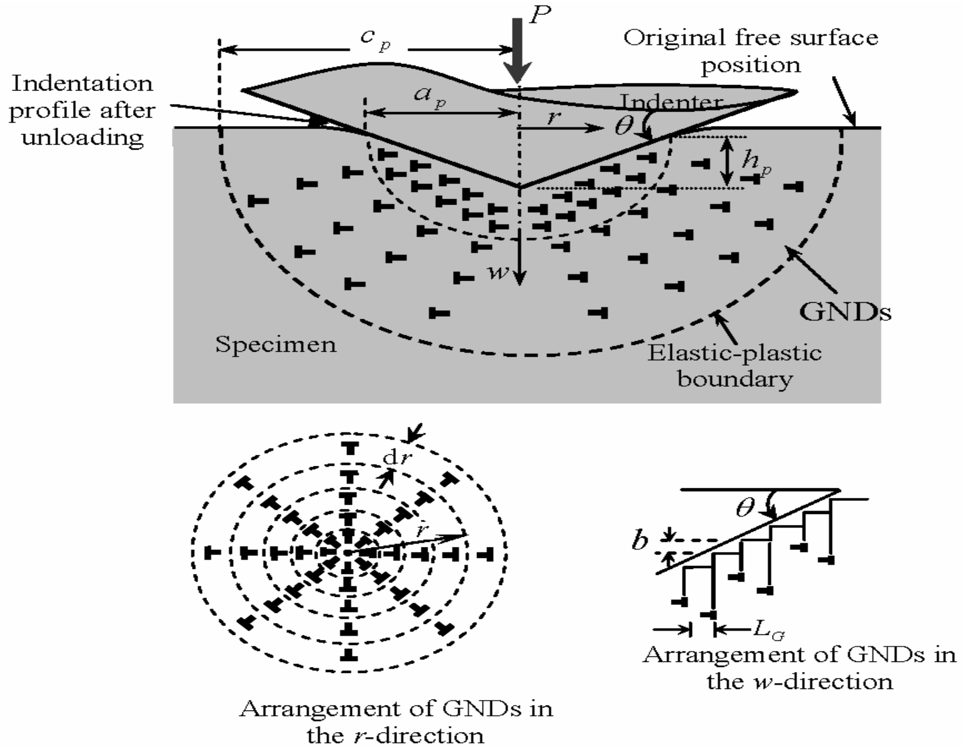


***Figure 6.*** *Illustration for the generation of GNDs due to ISE.*

Consider now the indentation by a rigid cone, as indicated in Figure 6. One can assume that the density of GNDs ($\rho_{GND}$) is integrated by the geometry of the indenter and the indentation is accommodated by circular loops of GNDs with Burgers vectors normal to the plane of the surface. One can use the hardness models [11] to determine the density of GNDs evolved under a conical/pyramidal indenter. As the indenter is forced into the surface of a single crystal, GNDs are required to account for the permanent shape change at the surface. Of course, SSDs, not shown in Figure 6, would also be created and they would contribute to the deformation resistance. The indentation profile in the unloaded configuration when using conical/pyramidal indenters can be described by (see Figure 6) [11]:

$$w(r) = h_p - r\,tan\,(\theta)\,, \text{where, } 0 \le r \le a_p \tag{11}$$

It is assumed that the dislocation evolution during indentation is primarily governed by a large hemispherical volume V that scales with the contact radius $a_p$ around the indentation profile (see Figure 6). However, the GNDs reside inside a plasticity zone which can be viewed as extending to a radius $c_p$ to the outermost dislocation emanated from the indent core. Therefore, the size of the plastic zone, $c_p$ , underneath the indenter is larger than the contact radius, $a_p$ , such that $c_p = f\,a_p$ where f > 1. Now, one can calculate the GND density ($\rho_{GND_s}$) using the following relation [11]:

$$\rho_{GND_s} = \frac{\lambda}{V} \tag{12}$$

Where λ is the total length of dislocation loops and V is the storage volume.

$$\lambda = \frac{\pi\, a_p\, h_p}{b} f^2 \tag{13}$$

One can then assume that all the injected GND loops remain within a hemispherical volume V of radius $c_p$, such that:

$$V = \frac{2}{3}\,\pi {c_p}^3 = \frac{2}{3}\,\pi f^3 {a_p}^3 \tag{14}$$

By substituting equations, (13), (14) in equation (12), the density of GNDs can be determined as follows:

$$\rho_{GND_s} = \frac{3\tan^2\theta}{2\,f\,b\,h_p} \tag{15}$$

The density of GNDs ($\rho_{GND}$) estimated from the hardness measurement changes exponentially as a function of deformation until to reach saturation as shown in Figure 7.

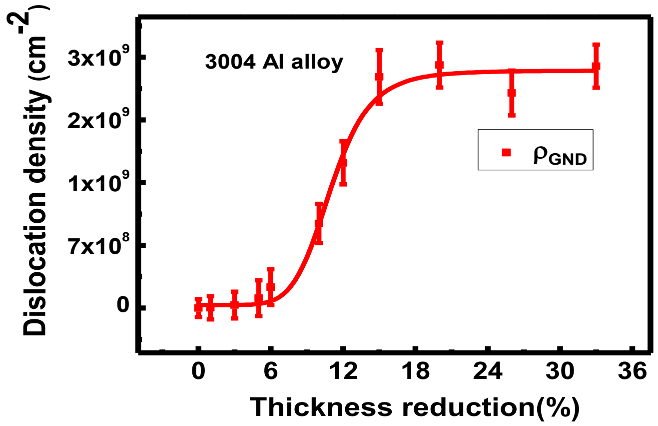


***Figure 7.*** *The density of GNDs ($\rho_{GND}$) as a function of thickness reduction for 3004 Al-alloy obtained by Vickers hardness measurement.*

In Figure 8, the value of total dislocation density ($\rho_t$) at 0.00 % thickness reduction is $4.9\times10^9$ cm$^{-2}$ and it varies to reach its maximum value, $1.42\times10^{10}$ cm$^{-2}$ , at 15.00 % thickness reduction, whereas, the density of GNDs ($\rho_{GND}$) value at 0.00 % thickness reduction is 0.00 and it varies to reach its maximum value $2.6\times10^9$ cm$^{-2}$ at the same thickness reduction. The density of SSDs is ($\rho_{SSD}$) = $4.9\times10^9$cm$^{-2}$.

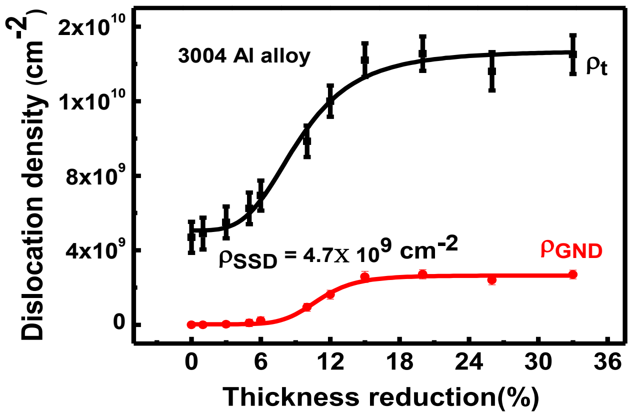


***Figure 8.*** *The dislocation density ($\rho_t$) and ($\rho_{GND}$) as a function of thickness reduction for 3004 Al-alloy obtained by Vickers hardness measurement.*

### *3.3.2. Studying the Behaviour of GNDs at Different Depth Stages*

The hardness of the material increases when the size of the deformed region decreases (as shown in Figure 9), this is known as the indentation size effect (ISE). The indentation depth (h) is related to the density of GNDs as cleared in equation (15) and density of SSDs as given by the following relation [12]:

$$\rho_{SSD} = \frac{3 \tan^2\theta}{2 f^3 b h^*} \quad (16)$$

Where, (θ) is the angle between the surface of the material and the surface of the indenter, (f) is a correction factor for the size of the plastic zone and ($h^*$) is a characteristic length that relies on the shape of the indenter [12]. In this work θ= 22°, f= 1.9 and b= 0.286 nm.

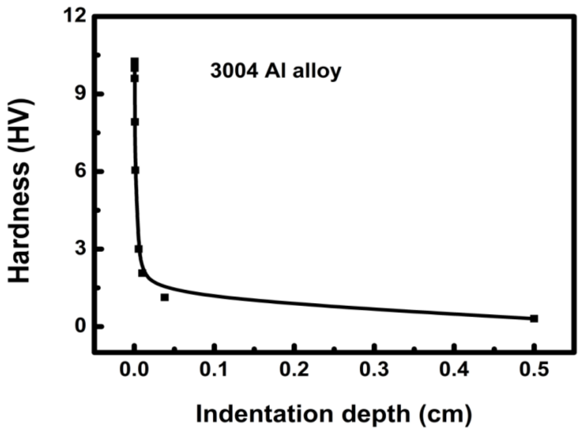


***Figure 9.*** *The hardness ($H_{GND}$) as a function of the indentation depth for 3004 Al-alloy obtained by Vickers hardness measurement.*

The authors made and developed a model to describe a simple expression to relate hardness with indentation depth as follows [11,12]:

$$(\frac{H}{H_0})^2 = 1 + h^* (\frac{1}{h}) \quad (17)$$

Where, H is the hardness for a given depth of indentation, h; $H_0$ is the hardness in the limit of infinite depth (bulk hardness) and (h*) is a characteristic length and is equal to $9 \times 10^{-4}$cm as indicated in Figure 10. The indentation depth decreases as function of thickness reduction as indicated in Figure 11.

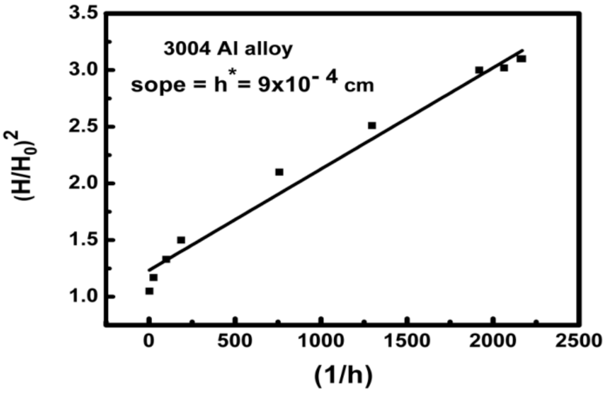


***Figure 10.*** *The ratio* $(\frac{H}{H_0})^2$ *versus* $(\frac{1}{h})$ *for 3004 Al-alloy obtained by Vickers hardness measurement.*

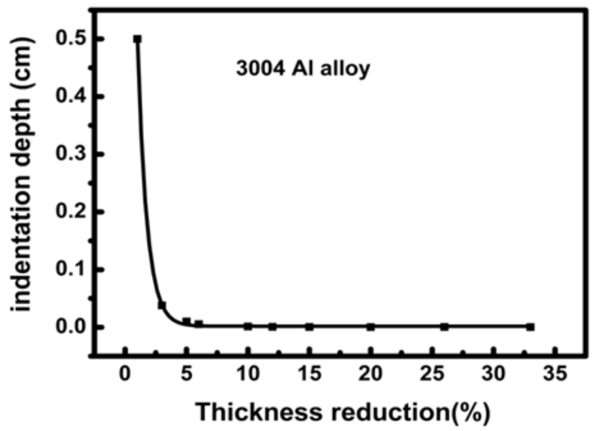


***Figure 11.*** *The indentation depth as a function of thickness reduction for 3004 Al-alloy obtained by Vickers hardness measurement.*

### *3.3.3. Estimation of the Density of Defects*

On the other side, the defect density ($\rho^-$) and the dislocation density ($\rho$) of the tested samples were calculated and the relation between them is given by:

$$\rho^{-}(\mathrm{cm}^{-3}) = \frac{\rho(cm^{-2})}{b} \quad (18)$$

The total defect density ( $\rho_t^-$) is increased as a function of degree of deformation as shown in Figure 12. Whereas the defect density of GNDs ($\rho_{GND}^-$) exhibit the same behavior as shown in Figure 13.

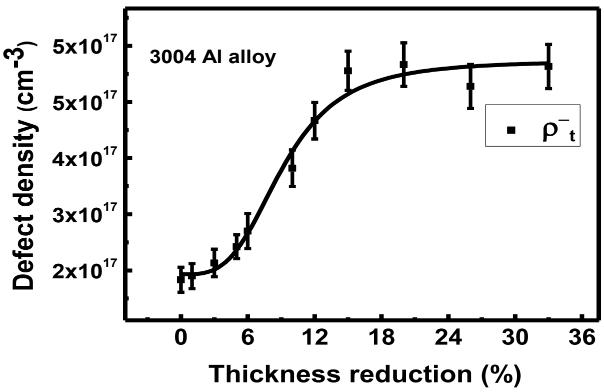


***Figure 12.*** *The total defect density ( $\rho_t^-$) as a function of thickness reduction for 3004 Al-alloy obtained by Vickers hardness measurement.*

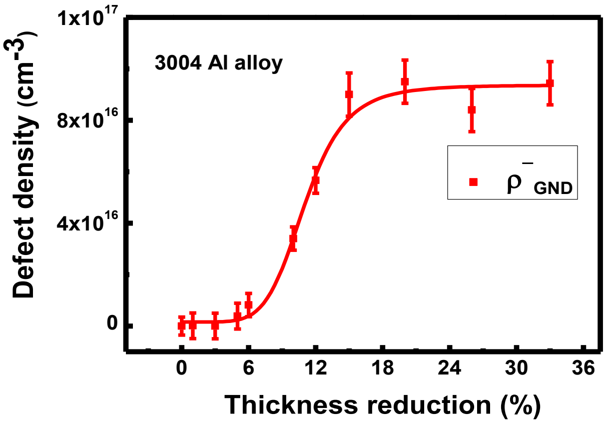


***Figure 13.*** *The defect density ($\rho_{GND}^-$ ) as a function of thickness reduction for 3004 Al-alloy obtained by Vickers hardness measurement.*

The value of total defect density ( $\rho_t^-$ ) at 0.00 % deformation is $1.65 \times 10^{17}$ $cm^{-3}$ and it varies to reach its maximum value, $5 \times 10^{17}$ $cm^{-3}$, at 15.00 % deformation, whereas, the defect density of GNDs ($\rho_{GND}^-$) value at 0.00 % deformation is 0.00 and it varies to reach its maximum value $9 \times 10^{16}$ $cm^{-3}$ at 15.00 % deformation. The defect density of SSDs is ($\rho_{SSD}^-$) = $1.65 \times 10^{17} cm^{-3}$. These variations were represented in Figure 14.

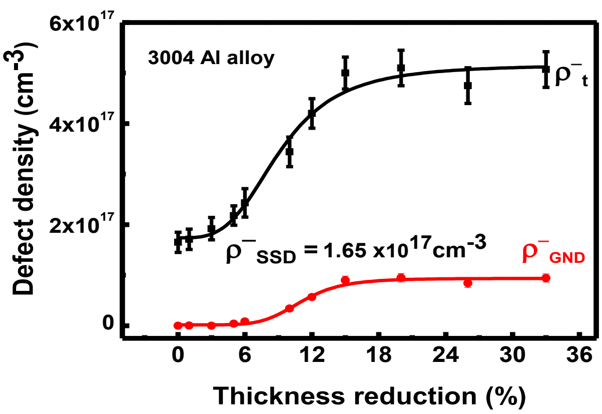


***Figure 14.*** *The defect density ($\rho_t^-$) and ($\rho_{GND}^-$) versus thickness reduction for 3004 Al-alloy obtained by Vickers hardness measurement.*

### 3.4. Determination of Mean Crystallite Size and Micro-Strain

The mean crystallite size is determined from this relation [13-14]:

$$l = \frac{(H_t - H_f)}{(H - H_f)} L_+ \quad (19)$$

Where (l) is the mean crystallite size, $H_f$ is the Vickers hardness value for the annealed sample, $H_t$ is the Vickers hardness value for the dislocation saturated sample, H is the Vickers hardness value at any thickness reduction value and $L_+ = 0.15$ μm is the diffusion length of the particle inside aluminum alloy.

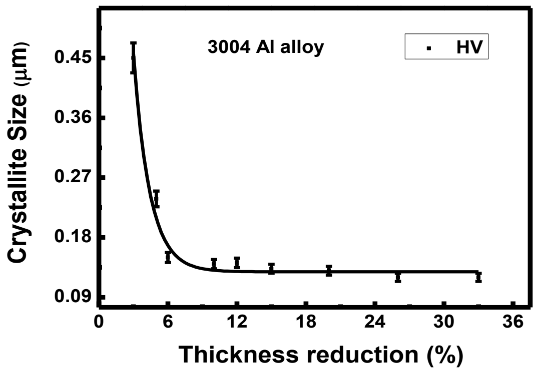


***Figure 15.*** *The mean crystallite size as a function of thickness reduction for 3004 Al-alloy obtained by Vickers hardness measurement.*

In Figure 15, the mean crystallite size of the investigated samples is decreased exponentially as a function of deformation which is decreased from 3.00 % thickness reduction at 0.45 μm to 10.00 % thickness reduction at 0.14 μm and becomes approximately constant above this value. On the other hand, the residual micro-strain $< \varepsilon^2 >^{\frac{1}{2}}$, is given by Vickers hardness measurement as a variation of (ρ) is calculated by using the given equation as follows [1]:

$$< \varepsilon^2 >^{1/2} = \frac{\rho \, L \, b}{3\sqrt{2\pi}} \quad (20)$$

The micro-strain is increased exponentially as a function of thickness reduction from $1.9 \times 10^{-4}$ at 0.00% thickness reduction to the value $6.9 \times 10^{-4}$ at 12.00% and it becomes approximately constant above this value as shown in Figure 16.

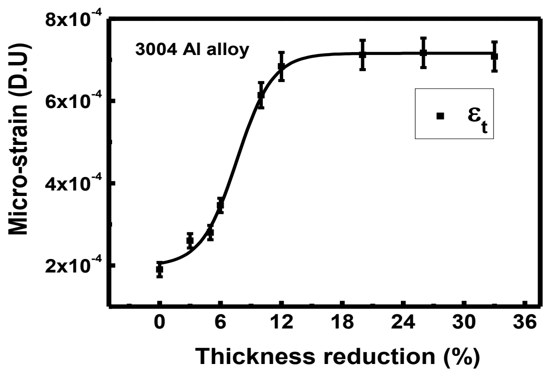


***Figure 16.*** *The micro-strain as a function of thickness reduction for 3004 Al-alloy obtained by Vickers hardness measurement.*

### *3.5. Estimation of the Flow Stress*

The term flow stress is used to describe the stress necessary to continue deformation at any stage of plastic strain. It is related to the square root of the dislocation density as follows [15]:

$$\sigma = \alpha\, Gb\sqrt{\rho} \tag{21}$$

The total flow stress ($\sigma_t$) of the tested samples and the flow stress as a result of Geometrically Necessary Dislocations, GNDs, ($\sigma_{GND}$) increase exponentially as a variation of degree of deformation or thickness reduction whereas the flow stress as a result of Statically Stored Dislocations, SSDs, is ($\sigma_{SSD}$) = 25.5 Mj/m$^3$ as shown in Figure 17, Figure 18, Figure 19.

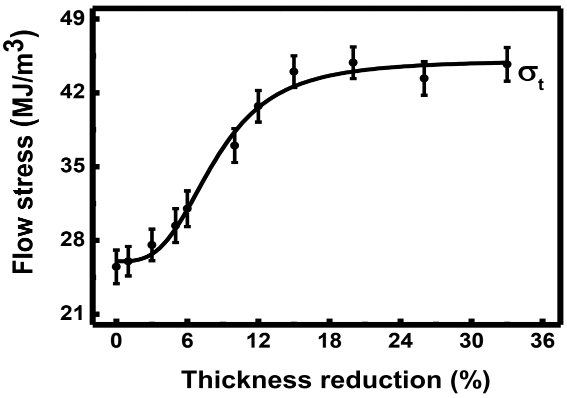


***Figure 17.*** *The total flow stress ($\sigma_t$) as a function of thickness reduction for 3004 Al-alloy obtained by Vickers hardness measurement.*

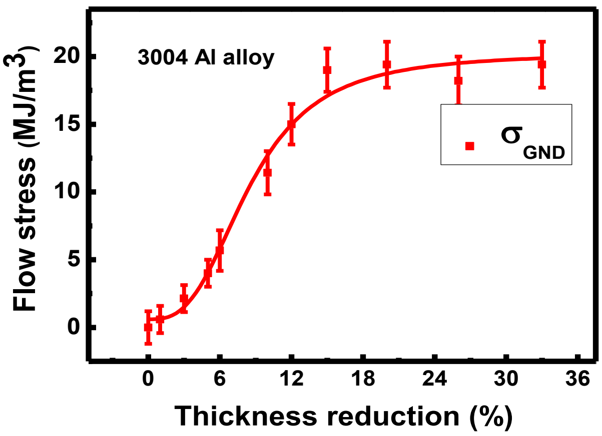


***Figure 18.*** *The flow stress ($\sigma_{GND}$) as a function of thickness reduction for 3004 Al-alloy obtained by Vickers hardness measurement.*

In Figure 19, the total flow stress ($\sigma_t$) of the tested samples increases from 25.5 Mj/m$^3$ at 0.00% thickness reduction to 44 Mj/m$^3$ at 15.00% thickness reduction , whereas the flow stress as a result of Geometrically Necessary Dislocations ,GNDs, ($\sigma_{GND}$) increases from 0.00 at 0.00% thickness reduction to 19 Mj/m$^3$ at 15.00% thickness reduction and they kept constant above 15.00% thickness.

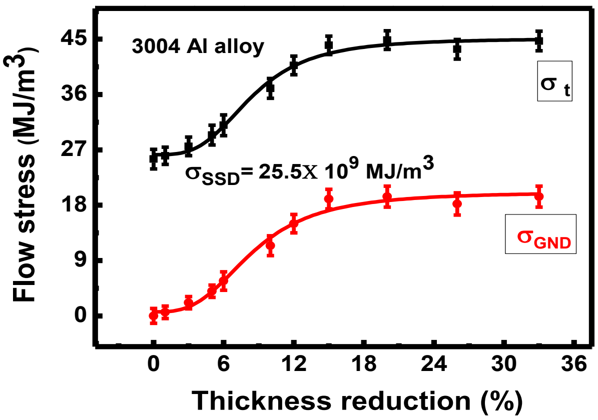


***Figure 19.*** *The flow stress ($\sigma_t$) and ($\sigma_{GND}$) as a function of thickness reduction.*

### *3.6. Estimation of the Stored Energy*

On these collections, the stored energy should be a sensitive factor of crystallite size, considerably at low strains. Both the stored energy and the manner in which the stored energy is released depend significantly on the domain size. The dislocation density ρ has direct relation with the stored energy (E) as follows [15]

$$E= \alpha\, Gb^2\, \rho \tag{22}$$

The total stored energy ($E_t$) of the tested samples and the stored energy as a result of, GNDs, ($E_{GND}$) increase exponentially as a variation of degree of deformation or thickness reduction whereas the stored energy as a result of Statically Stored Dislocations, SSDs, is ($E_{SSD}$) = 50 Kj/$m^3$ as indicated in Figure 20, Figure 21, Figure 22).

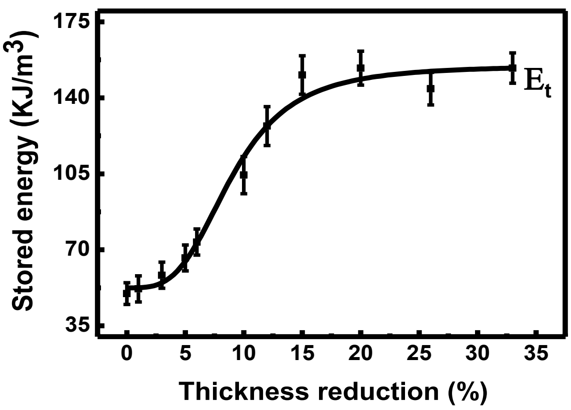


***Figure 20.*** *The total stored energy ($E_t$) versus thickness reduction.*

In Figure 22, the total stored energy ($E_t$) of the tested samples increases from 49.8 Kj/$m^3$ at 0.00% thickness reduction to 150.5 Kj/$m^3$ at 15.00% thickness reduction , whereas the stored energy as a result of ,GNDs, ($E_{GND}$) increases from 0.00 at 0.00% thickness reduction to 27.4 Kj/$m^3$ at 15.00% thickness reduction and they kept constant above 15.00% thickness reduction.

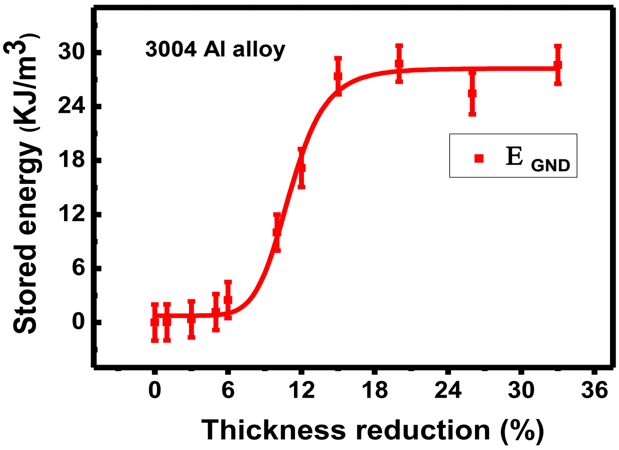


***Figure 21.*** *The stored energy ($E_{GND}$) as a function of thickness reduction.*

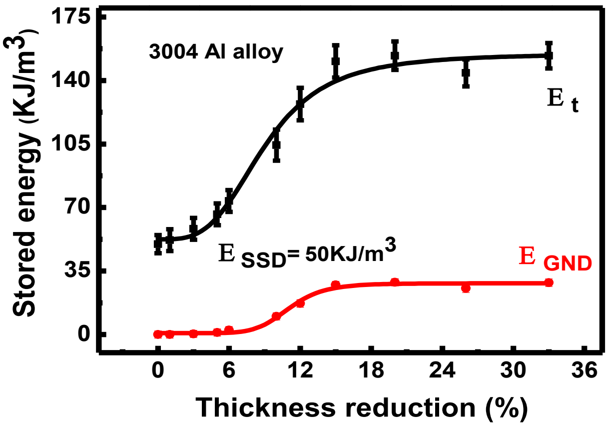


***Figure 22.*** *The stored energy ($E_t$) and ($E_{GND}$) versus thickness reduction.*

## 4. Conclusion

According to the results and discussions obtained from hardness measurements the following items we can conclude:

I Novel methods were successfully applied to calculate the total hardness, the hardness of GNDs and hardness of SSDs in addition to the mean crystallite size and micro-strain.

II The total hardness and the hardness of GNDs exhibit an exponential increase in the range from 0.00% thickness reduction to 15% thickness reduction and they become constant above this value. The hardness due to (SSD) is H_SSD =13.49 HV.

III The mean crystallite size exhibit an exponential decrease due to the grain size reduction due to the deformation effects

IV The defect density, micro-strain, flow stress and stored energy exhibit the same behaviour and they behave like the dislocation density due to the increased number of dislocations in the slipping planes as a result of the dislocation interactions.

## Conflicts of Interest

The authors declare that there is no conflict of interest regarding the publication of this article.

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