Effect of sputtering target's grain size on the sputtering yield, particle size and coercivity (Hc) of Ni and Ni20Al thin films

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Abstract. Researches on magnetic thin films concentrated mainly on optimizing the sputtering parameters to obtain the desired thin film's properties. However, the effect of the sputtering target's properties towards the thin film's properties is not well established. This study is focused on analysing the effect of sputtering target's grain size towards the sputtering yield, particle size and the magnetic coercivity (Hc) of thin film. Two sets of sputtering targets; pure Ni (magnetic) and Ni20Al (at.%) (non-magnetic) were prepared. Each target has 2 sets of samples with different grain sizes; (a) 30 to 50µm and (b) 80 to 100µm. Thin films from each target were sputtered onto glass substrates under fixed sputtering parameters. The initial results suggested that the sputtering target's grain size has significant effect on the thin film's sputtering yield, particle size and Hc. Sputtering target with smaller grain size has 12% (pure Ni) to 60% (Ni20Al) higher sputtering yield, which produces thin films with smaller particle size and larger Hc value. These initial findings provides a basis for further magnetic thin film research, particularly for the seed layer in hard disk drive (HDD) media, where seed layer with smaller particle size is essential in reducing signal–to-noise ratio (SNR).

1. Introduction

Research of magnetic thin films has been of deep interest due to its usage in various applications, such as sensors, actuators and magnetic recording hard disk drives (HDD). Recent developments on social media and cloud data storage have further increased the demands for higher data storage capabilities of HDD [1-2]. Hence, extensive studies have been done to increase the storage capabilities of HDD [3]. One approach is to reduce the particle size of the recording layer to increase Signal-to-Noise ratio (SNR). The recording layer is grown epitaxially on a seed layer, which consist of non-magnetic material. Hence changes on the seed layer's particle size would directly impact the particle size of the recording layer leading to better SNR and higher storage capabilities.

Studies on the effect of sputtering parameters such as deposition time [4-7], sputtering power [8-10], type of substrate [8], substrate temperature [11-13], and post-sputter annealing temperature

[14-15] on the deposited thin film properties have been done extensively. However, the relation between the sputtering target's mechanical properties, such as grain size and the thin film's properties is not well established.

Previous study on NiAl alloy have been shown it to be a suitable seed layer candidate, where NiAl seed layer were able to reduce the particle size of Ru intermediate layer and CoCrPt recording layer, leading to a higher SNR [3-4,16]. In this study, the effect of grain size of pure Ni and NiAl sputtering targets on the sputtering yield, particle size and coercivity (Hc) of deposited thin films was investigated. For each alloy, 2 sets of sputtering targets with different grain sizes; (a) 30 to 50μm and (b) 80 to 100μm were fabricated. Thin films from each sputtering target were deposited onto glass substrates under fixed sputtering parameters. The properties of the thin films were analysed to determine the correlation between grain sizes of sputtering target on thin films properties.

2. Method

In this study, magnetic pure Ni and non-magnetic Ni20Al (20at% of Al) materials were chosen to determine if magnetic saturation (Ms) has any effect on the thin film formation. Pure Ni and Ni20Al ingots were fabricated using Vacuum Induction Melting (VIM) equipment. These ingots were then hot rolled to 50% of its original thickness at 1200°C, followed by 2 hours of annealing before cooled in air. Smaller samples were then cut from each ingot and cold rolled to 70% to 80% of its original thickness, followed by heat treat ment at temperatures ranging from 600°C to 1250°C. The heat treatment time ranges from 60 minutes to 390 minutes, followed by water quenching.

The heat treatment process is controlled in order to produce two batches of bulk samples with different ranges of grain size; (A) 30 to 50μm and (B) 80 to 100μm. Samples were then polished and etched, before analysed using the scanning electron microscope (SEM) in order to determine the grain size.

Using a DC magnetron sputtering equipment, the pure Ni and Ni20Al bulk samples (from now on will be referred to as *sputtering targets*) were deposited on glass substrates. The sputtering parameters were fixed; base pressure = $9x10^{-5}$torr, working pressure = $3x10^{-1}$torr, Argon gas flow = 20 to 30 sccm, current = 28 to 33 Watt, substrate temperature = 250°C, target-substrate distance = 3cm, and sputtering time = 60 minutes. The grain size of the sputtering targets was determined using the linear intercept method, while the particle size of the thin film samples was determined using Image J software. The coercivity (Hc) of both sputtering targets and thin films were determined using a vibrating-sample-magnetometer (VSM).

3. Result and discussion

The grain sizes of all bulk samples produced are shown in Table 1. The magnetic saturation of all samples is also listed in this table. The result confirmed that the pure Ni alloy produced in this study was magnetic, while Ni20Al exhibited non-magnetic properties. The Ms value in Ni20Al (small grains) and (large grains) reduced significantly as compared to that in pure Ni (small grains) and (large grains) alloys. It is believed that higher Al contents reduces the Ms of the Ni, as free electrons from Al 4s orbital fills up the empty spaces in Ni 3d orbital, reducing the magnetic moment [17].

Table 1. Grain size and magnetic saturation (Ms) of sputtering target.

Sputtering targets	Grain size (μm)	Magnetic saturation, Ms (emu/cc)
Pure Ni (small grain)	54	642.2
Pure Ni (large grain)	102	511.9
Ni20Al (small grain)	32	3.8
Ni20Al (large grain)	83	1.7

3.1 Effect of grain size on sputtering yield

In order to determine the effect of grain size towards the sputtering yield, the cross section of the thin film were observed using SEM. Sputtering duration was fixed on all samples; hence thicker samples are classified as having higher sputtering yield. For this study, the sputtering yield is defined as deposited thin film per minute and is summarized in Table 2. The thin film thickness of each sample is plotted in Figure 1.

For both samples, sputtering targets with smaller grain size produces thicker films under the same duration, i.e. 12% (pure Ni) to 60% (Ni20Al). This result is believed to be attributed to the increased bombardment of incident ions on the faceted planes (Figure 2). Smaller grains will erode faster from the sides, producing more ejected atoms compared to larger grains, hence contributing to the formation of thicker thin films.

Table 2. Effects of grain size of sputtering target–on deposited thin film thickness and sputtering yield. Sputtering duration was set at 60 minutes.

Sample	Sputtering target's grain size (μm)	Thin film thickness (nm)	Sputtering yield (nm/min)
Pure Ni (small grain)	54	391	6.5
Pure Ni (large grain)	102	344	5.7
Ni20Al (small grain)	32	13384	223.1
Ni20Al (large grain)	83	8335	138.9

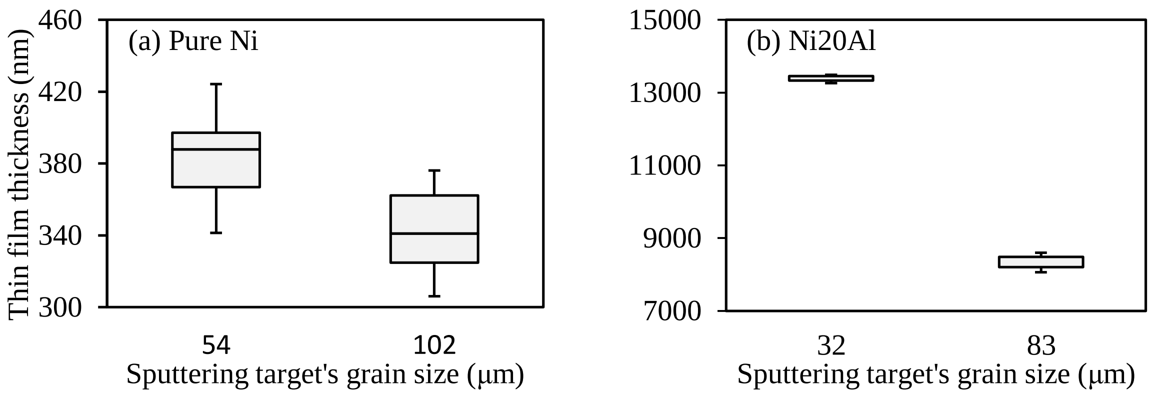

Figure 1. Box plot represents the thin film thickness deposited from (a) Pure Ni and (b) Ni20Al sputtering targets with different grain size for 60 minutes.

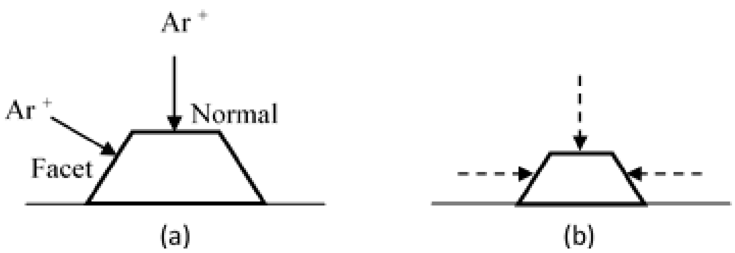

Figure 2. Sputtering sequence on grains;
(a) Ar $^{+}$ bombardment on normal and faceted plains of grains.
(b) Continuous erosion from both normal and faceted planes hastens the reduction of the cross-sectional area of the grain.

Relation between the times required to completely erode a crystallite with its grain size has been summarized previously by Michaluk [18];

$$t = \frac{A^2}{2k} \quad (1)$$

where;
t = time to erode crystallite
A = crystallite area perpendicular to incident beam
k = constant of proportionality
Results obtained in this study are in line with the above correlation.

The result also showed that non-magnetic Ni20Al has higher sputtering yield (>80%) as compared to magnetic pure Ni. In DC magnetron sputtering system, a magnet is equipped in order to increase the ionizing effect on Ar gas to create Ar plasma. The magnetic flux from the rotating magnet traps the electron in a magnetic field. Higher concentration of trapped electrons will produce more plasma, hence inducing more bombardment on target's surface which leads to more ejected atoms per minute. In this research, the magnetic flux from pure Ni targets obstructed the flux from the rotating magnet, therefore effecting the ionization of Ar gas which reduced the formation of Ar plasma in the sputter chamber. Less bombardment during sputtering has lowered the sputtering yield of pure Ni targets.

3.2 Effect of grain size on particle size

Figure 3 until Figure 6 displayed the plain-view of grain and particle images of the sputtering target and sputtered thin film for pure Ni and Ni20Al, respectively. The result showed that, for both alloys, sputtering targets with larger grain size deposited thin films with smaller particle size. Pure Ni thin films particles from pure Ni (large grain) sputtering target are 5.6% smaller than thin film particles from pure Ni (small grain) sputtering targets. Similarly, Ni20Al thin films particles from Ni20Al (large grain) sputtering target are 51.8% smaller than thin film particles from Ni20Al (small grain) sputtering target.

From Table 1, we understand that sputtering targets with smaller grains have higher sputtering yield, hence more adatoms are deposited on the substrate, compared to sputtering targets with larger grains. The rate of coalescence between adatoms to form islands will be higher, leading to the formation of larger particles.

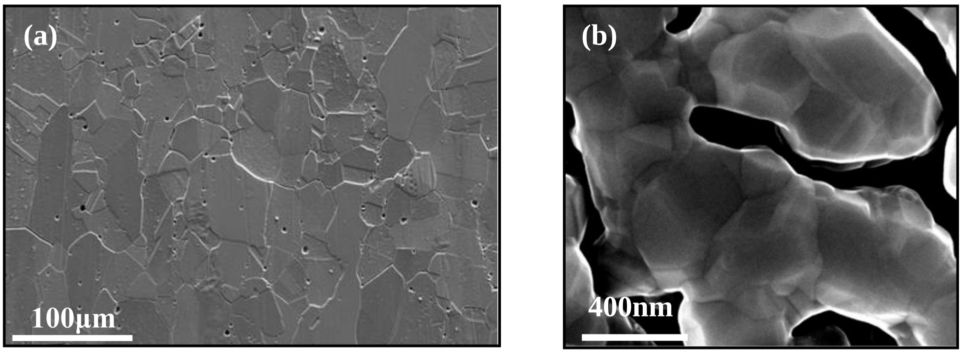

Figure 3. Plane-view SEM images of (a) Pure Ni sputtering target (small grain), Grain size = 54µm and (b) Deposited pure Ni thin film, Particle size = 196nm.

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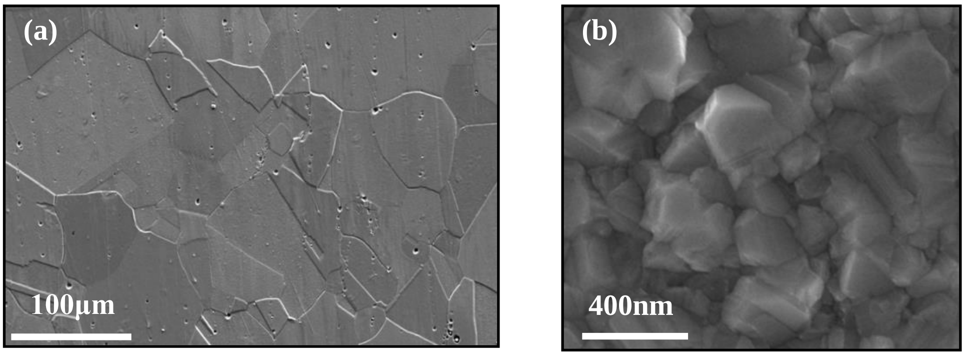

Figure 4. Plane-view SEM images of (a) Pure Ni sputtering targets (large grain), Grain size = 102μm and (b) Deposited pure Ni thin film, Particle size = 185nm.

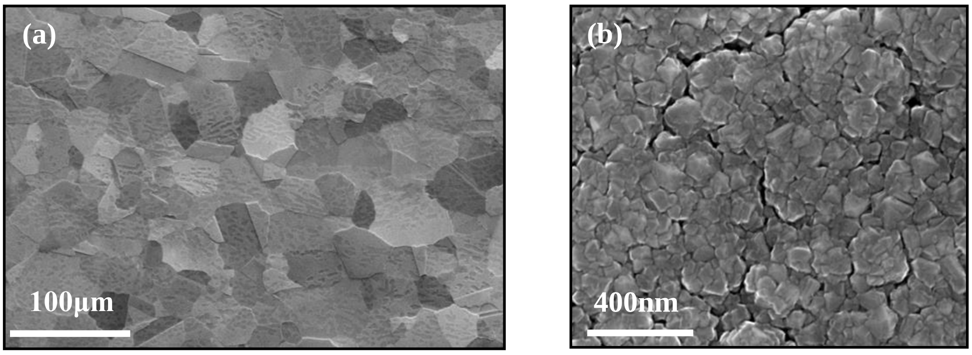

Figure 5. Plane-view SEM images of (a) Ni20Al sputtering targets (small grain), Grain size = 32μm and (b) Deposited Ni20Al thin film, Particle size = 56nm.

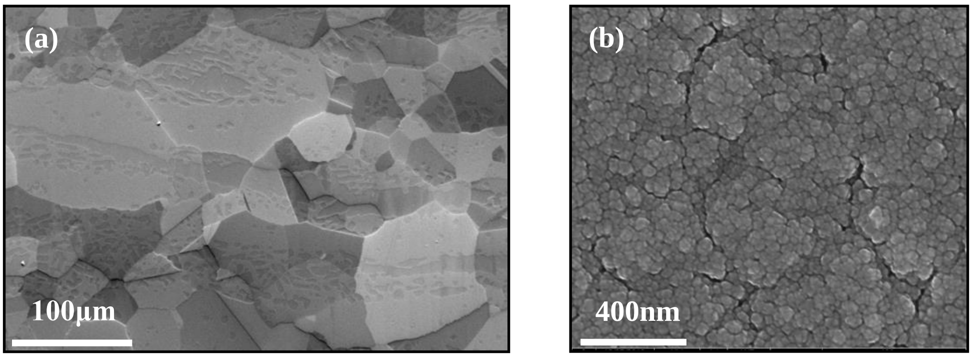

Figure 6. Plane-view SEM images of (b) Ni20Al sputtering targets (large grain), Grain size = 83μm and (b) Deposited Ni20Al thin film, Particle size = 27nm.

3.3 Effect of grain size on magnetic coercivity (Hc)

Table 3 correlates the magnetic coercivity (Hc) of thin film with its particle size. The result showed that thin films with smaller particles have lower Hc value. This result suggest that the particle of the thin films are multi-domains. Particles below its critical size are single-domain, where the magnetic moment of the particle increases with increased particle size. Since stronger field is required for magnetization reversal, therefore Hc value also increases with increased particle size.

However, above the critical size, the competition between the increasing magneto-static energy and the domain-wall energy favors domain-wall formation and the single-domain particle splits into multi-domain. The magnetization processes in a multi-domain sample are accomplished by domain-wall motion, which is normally completed in weak applied fields, and then by magnetization rotation. This will lead to a decrease in Hc (due to splitting of the magnetic domains), which therefor exhibits a peak at the critical single-domain size. Above this limit, Hc reduces with increased particle size.

Table 3. Correlation between thin film particle sizes with coercivity, (Hc) value.

Sample	Sputtering target's grain size (μm)	Thin film particle size (μm)	Thin film coercivity (Hc)
Pure Ni (small grain)	54	196	103.7
Pure Ni (large grain)	102	185	107.4
Ni20Al (small grain)	32	56	41.4
Ni20Al (large grain)	83	27	53.2

4. Summary

The effect of the grain size of pure Ni and Ni20Al sputtering targets towards the sputtering yield, particle size and coercivity (Hc) of thin films deposited by DC magnetron sputtering on glass substrates were examined. SEM was used to observe of the grain size of sputtering targets, particle size and thickness of thin films. Result showed that sputtering targets with smaller grain size has higher sputtering yields and produced thin films with larger particle. This is true for both magnetic and non-magnetic targets. However, magnetic pure Ni has lower sputtering yield as flux from the magnet was obstructed by the magnetic flux from pure Ni targets, effecting the ionization of Ar gas therefore reducing the bombardment of Ar ions on sputtering targets. VSM analysis revealed that thin films with larger particles have lower coercivity (Hc), suggesting that particle of thin films consists of multi-domains. The findings of this study can be used as a basis to optimize the particle size and magnetic coercivity of seed layer in HDD.

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