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Investigation of Thickness Effect on Structural and Magnetic Properties of Ni Thin Films for Some Applications

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Abstract

In this study, it was investigated the effect on the structural, and magnetic properties dependent on the thickness of the Nickel films grown on MgO (100) substrates by the molecular beam epitaxy at a high vacuum. The structural and magnetic properties were examined by using X-ray diffraction and ferromagnetic resonance techniques. The X-ray diffraction and X-ray reflectivity measurements showed that Ni films grew in (200) orientation with tiny surface roughness. Experimental ferromagnetic resonance data showed that all samples had in-plane easy axis from out-of-plane measurements and fourfold anisotropy from in-plane measurements. Additionally, conditions under which Ni/MgO (100) films grew epitaxially were also observed. In this study, where the minimum thickness required for some applications to exhibit both magnetic properties and form the crystal structure of Ni thin films is determined, the importance of film thickness in terms of applications is emphasized and the minimum thickness condition is determined in terms of some applications.

Keywords: Ferromagnetic Resonance, Magnetic Anisotropy, Ni Thin Films, Spintronics, Thin Film Applications.

1. Introduction

Recently, significant developments in mechanical, biomedical, and spintronic technologies have attracted attention [1–5]. Studies in this field have gained great importance, and this situation has increased the need for new technologies and materials for use in various biomedical applications. Nickel (Ni) thin films, which have an important place among these materials because of their wide range of uses in various technological and scientific applications, attract the attention of researchers due to their properties such as electrical and magnetic properties, corrosion resistance, chemical stability, and mechanical durability.

Ni-based thin films are widely used in microelectromechanical systems (MEMS) due to their high hardness and good mechanical strength properties [6]. Such films are frequently used to coat moving parts in MEMS devices and the production of micro-scale actuators. They are also ideal materials for applications requiring friction and corrosion control due to their high corrosion resistance and surface hardness [7].

On the other hand, Ni-based thin films are also indispensable materials for spintronic applications, which are a field that aims to develop information processing and storage technologies using the spin property of electrons [8- 10]. In studies of this field, it is seen that Ni-based thin films are frequently used as key materials in spintronic devices such as magnetic tunnel junctions (MTJs) and spin valves [11]. These films are also quite useful in optimizing the performance of spintronic devices due to their magnetic anisotropy and low magnetic damping properties [12,13]. In addition, Ni is a magnetic material with a high Curie temperature (T_C) and is preferred in spintronic device development because this feature allows the devices to operate in a wide temperature range [14].

Furthermore, thin films have a wide range of biomedical applications such as biomedical devices, surgical instruments, sensors (such as glucose sensors), magnetic resonance imaging (MRI), biocompatible coatings, human implants, and drug delivery systems [15–19]. On the other hand, it is important also to examine the behavior of these materials in biological environments since the possible interaction of Ni thin films with biological systems may cause a potential negative effect on health, such as Ni allergies and toxicity [20]. In addition, it can be accepted that the structure and thickness of the films are the determining parameters in the interaction of Ni thin films with biological systems.

In this context, Ni thin films of different thicknesses were prepared in the current study. It was aimed to investigate the magnetic and structural properties of Ni thin films of different thicknesses to increase their potential in some application fields such as magnetic storage devices, sensors, MEMS, biomedical devices, etc., and to create a background for these studies.

2. Materials and Methods

The Ni thin films were grown on magnesium oxide [MgO-(100)] substrates. The molecular beam epitaxy (MBE) thin film growth system was used and its base pressure was 7×10^{-10} mbar. Before growth, all MgO substrates were cleaned in acetone, methanol, and isopropanol using an ultrasonicator for 15 minutes. Additionally, all substrates were kept under thermal treatment for 1 hour at 475°C to remove the rest chemicals on the surface of the substrates and cooled down to room temperature (RT). The films were fabricated in different thicknesses (5.0 nm, 15 nm, and 30 nm) at a 2.022 nm/min deposition rate. After the growth procession, all Ni thin films were cooled down to the RT.

While the crystal structure and film thickness of the Ni thin films were examined by X-ray diffractometry (XRD), the magnetic properties were investigated by using an X-band JEOL series electron spin resonance (ESR) spectrometer with a microwave frequency of 9.8 GHz (JESFA 300) at the RT. The ferromagnetic resonance (FMR) measurements were carried out in both out-of-plane geometry (OPG) and in-plane geometry (IPG). During the measurement, the sample was rotated in the magnetic field, which was scanned between 0-2 Tesla.

The FMR technique is a very successful method used to analyze magnetic thin films' anisotropic behaviors [25]. This method can provide high precision, such as magnetic anisotropy and the size of magnetism [25–27]. This technique is based on magnetic resonance and is used to investigate the magnetic properties of samples that have one or more unpaired electrons. That is, the interaction of the magnetic moments of the electrons within the atom is examined. The magnetic moments behave like magnets when samples with unpaired electrons are placed in an external magnetic field. The spin values of unpaired electrons are split according to the allowed energy levels in this external field, and the Larmor frequency is directly proportional to the difference between these energy levels. Then, a microwave frequency is applied perpendicular to the field, and resonance occurs when this frequency is equal to the difference between the energy levels. While the frequency at which the resonance emerges corresponds to the resonance frequency, the magnetic field at which the resonance emerges corresponds to the resonance field. FMR experiments are performed using the ESR spectrometer. It is possible to determine the easy axis of magnetization since these experiments provide the opportunity to carry out a magnetic field to the sample at different angles. During the measurement, the interaction of the magnetic moments of the electrons in the sample is examined by using a microwave source in the wavelength range of 300 MHz-300 GHz.

3. Results and Discussion

Structural analysis of Ni/MgO (100) thin films was examined by XRD and X-ray reflectivity (XRR) measurements at the RT. Figure 1(a-c) shows the XRR oscillations of each sample and the simulation results obtained from the GlobalFit program used to analyze the XRR data [28]. The XRR fitting was made using the bulk density values of Ni while using the GlobalFit program. Perfect compatibility of the obtained fitting lines (red line) with experimental data (black empty circles) is important for the reliability of the results. The fact that the surface roughness obtained as a result of the fitting is below 1 nm is an important indicator of the growth of high-quality films (Table 1). Figure 1(d-f) shows the XRD patterns of all Ni/MgO (100) films. It is observed that only the thickest sample exhibits a distinct Ni (200) film peak [29]. Especially the fact that the thickest sample gives a distinct Ni (200) peak is an important indicator that the film thickness is effective in the formation of the crystal structure. Normally, Ni thin films are expected to oxidize when they come into contact with air from the moment they are removed from the system after preparation, and this oxide structure is detected by the oxide peak obtained in XRD measurements. However, no oxide peaks were found in the XRD patterns since XRD measurements were made immediately after the Ni film was prepared. Oxidation will continue as a slow and natural process once Ni is exposed to air, and it is known that this oxide layer that may form does not exceed 10 Å.

Table 1. The properties of Ni thin films.

Sample Name	Ni Thickness (nm)	Roughness (nm)
S1	5.0	0.420
S2	15	0.460
S3	30	0.152

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The FMR measurements were performed at RT to examine the magnetic properties of Ni thin films, (Figure 2). The measurements were made at both the IPG (by applying a magnetic field parallel to the sample plane) and the OPG (by applying a magnetic field perpendicular to the sample plane) for each sample. During the measurement, the sample is placed in the cavity between two magnets and rotated in one direction at determined angles, and the resonance fields occurring at each angle are read. As a result of the measurement, the angular change of the resonance fields of the samples is obtained at both OPG (Figure 2(a-c) and IPG (Figure 2(d-f)). As a result of the FMR measurements, the easy and hard axis of magnetization of these magnetic thin films can be determined, as well as information about the type of magnetization is obtained. The easy axis is defined as the direction in which the magnetic moments of magnetic thin films are easily aligned in the direction of the magnetic field in the presence of a magnetic field [25]. It can be seen that the OPG is a hard axis and the easy axis is in the IPG for each sample looking at out-of-plane (OOP) measurements. Therefore, it can be said that Ni thin films have an in-plane (IP) easy axis for this sample structure. In the easy axis direction, interfacial tensions between the substrate and the film and crystal structure properties affecting magnetic anisotropy are the determining factors in the magnetic properties of thin films [25, 30-32].

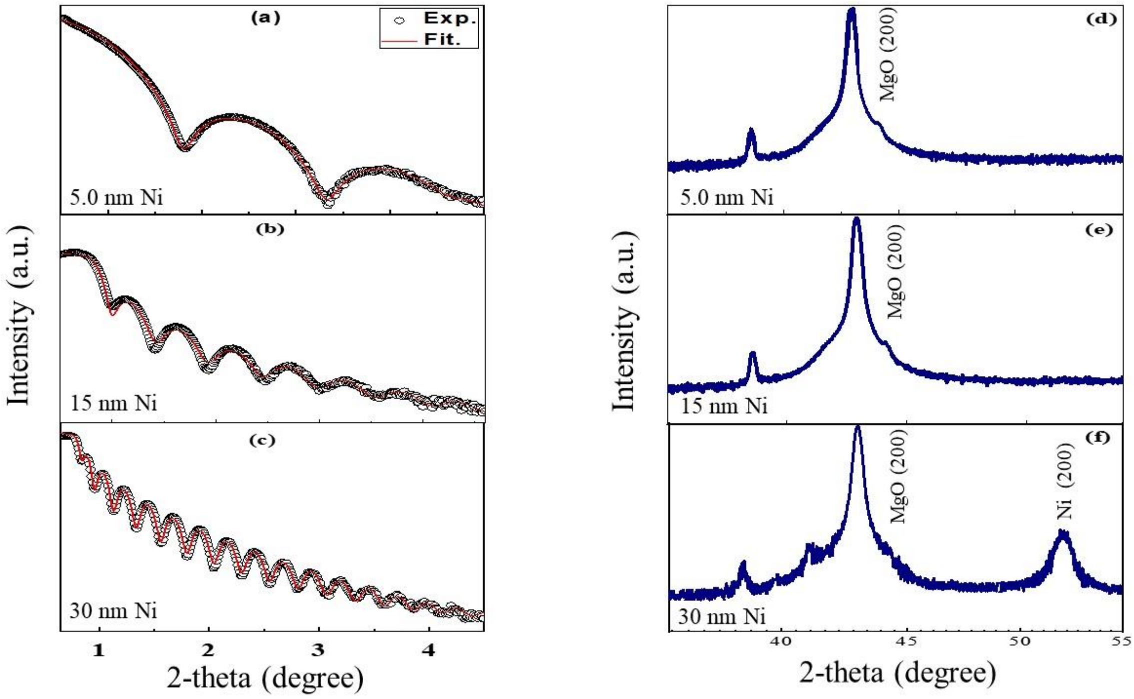

Figure 1. X-ray reflectivity (XRR) results of samples. The roughness of each film is 0.42 **(a)** for 5.0 nm Ni, 0.46 **(b)** for 15 nm Ni, and 0. 152 nm **(c)** for 30 nm Ni. The red line and the black empty circle represent fitting and experimental data, respectively. X-ray diffraction (XRD) measurement results of **(d)** 5.0 nm Ni, **(e)** 15 nm Ni, and **(f)** 30 nm Ni.

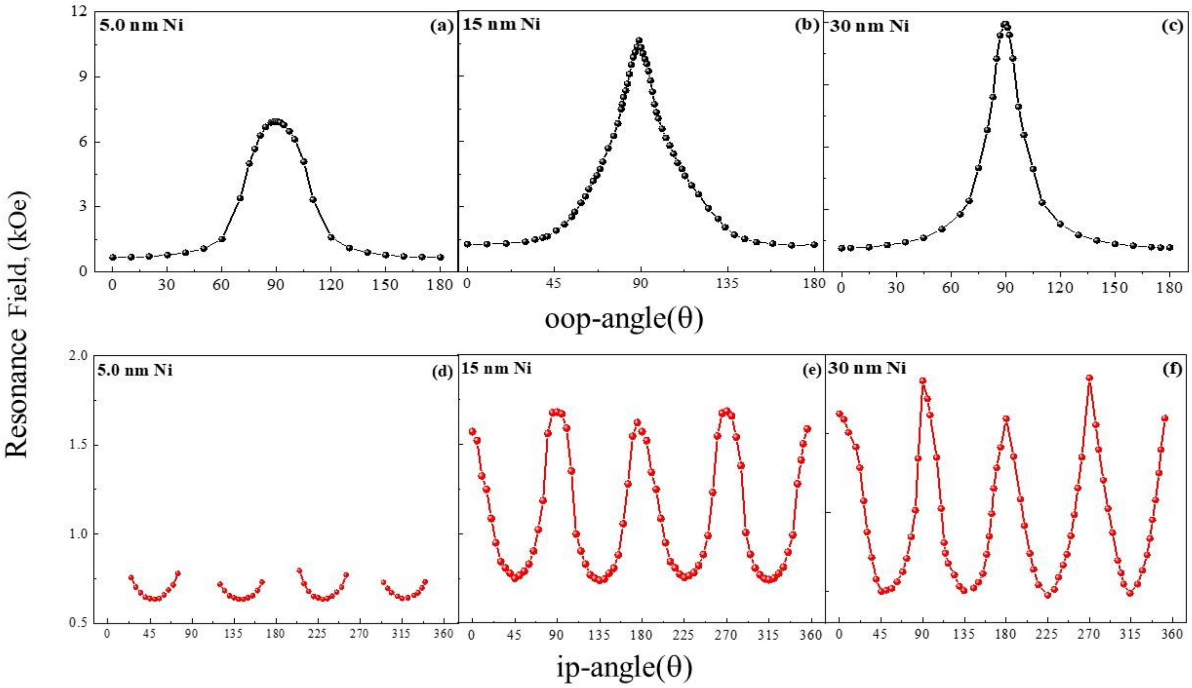

Figure 2. Ferromagnetic resonance fields of Ni thin films **(a)** for 5.0 nm Ni, **(b)** for 15 nm Ni, **(c)** for 30 nm Ni at the OPG. Ferromagnetic resonance fields of Ni thin films **(d)** for 5.0 nm Ni at the IPG, **(e)** for 15 nm Ni at the IPG, and **(f)** for 30 nm Ni at the IPG.

The measurements made in IPG are exhibited in Figure 2(d-f). The existence of fourfold anisotropy [33, 34] is seen for the S2 and S3 samples although the presence of a four-fold anisotropy is not evident for the thinnest Ni thin film. The substrates used in thin films are parameters that directly affect the physical properties of the films [35, 36]. The MgO substrates are among the substrates frequently used when preparing metallic thin films. The MgO (100) substrate used in this study has a cubic crystal structure. Ni has a face-centered cubic (fcc) crystal structure and is a suitable element to grow onto the surface of the MgO (100) substrate [37]. During the growth process, Ni atoms align in harmony with the crystal structure of the MgO substrate and enable the growth of an epitaxial film. Therefore, the MgO substrate, which has a certain crystallography, enables Ni films to grow in a certain crystallographic structure. The fourfold anisotropy observed from FMR measurements made in in-plane geometry in this study is a situation related to MgO substrate with four different growth orientations such as [100], [110], [111], and [001] [38]. As a result, the fact that Ni thin films have an IP easy axis according to OOP measurements and that they exhibit fourfold anisotropy according to IP measurements indicates that the samples grow in harmony with the crystallographic structure of the substrate and exhibit epitaxial growth [39, 40].

4. Conclusion

The effects of film thickness on Ni films' magnetic and structural properties have been investigated in this study. Ni thin films with different thicknesses have been deposited directly on the MgO (100) using the MBE system. The structural and magnetic properties have been examined using the XRD and the FMR techniques at RT. The cubic crystalline Ni films preferred growth orientation along the (200) plane and the roughness of each of the Ni films have been determined from the XRD and XRR measurements, respectively. Additionally, the easy axis direction and magnetic anisotropy behavior of Ni films have been identified from the IP and OOP-FMR measurements. As a result, it was observed that the crystal structure was formed more clearly for Ni films of over 15 nm, and higher quality epitaxial films were formed by following the crystal structure of the substrate.

Since the formation of the crystal structure after a certain thickness will provide high hardness and good mechanical strength properties, it will increase efficiency in MEMS applications. Because the thickness of the thin films prepared in spintronic applications has a significant effect on all properties, it is important in which thickness the thin film prepared exhibits both structural and magnetic properties. Otherwise, as the effects from that material will be lost in the presence of interlayers or other elements if the thickness remains below this limit, it is important to determine the lower thickness limit. Because the growth of the Ni layer below this thickness will not make any contribution to the structure and will not be effective in any of its magnetic properties. Lastly, for biological applications, in applications where mechanical strength is required such as surgical instruments and human implants, the formation of the crystal structure is necessary and it has been shown that this structure is formed after a certain thickness. In addition, in biological sensor applications, starting from the minimum thickness where the magnetic behavior of Ni is evident will provide results. For all these reasons, the preparation and use of Ni films in thicknesses where both crystal and magnetic structures are formed together stably are essential for some applications. Therefore, in application fields such as mechanical, biomedical, and spintronic technologies, it would be more appropriate to use Ni film thicknesses of min above 15 nm in terms of both efficiency and physical properties. The background created by this result obtained in this study in terms of Ni film applications is important in terms of preventing waste of time and labor.

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Author's Contributions

Perihan AKSU: Conceptualization, Methodology, Formal analysis and investigation, Validation, Writing-Reviewing and Editing, Writing- Original draft preparation.

Ethics

There are no ethical issues after the publication of this manuscript.

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