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高频用 Fe-Ni 基纳米软磁薄膜的制备与研究

Fabrication and Investigation of the Fe-Ni Based Nanostructured Soft-magnetic Film for High-frequency Application

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Fabrication and Investigation of the Fe-Ni Based Nanostructured Soft-magnetic Film for High-frequency Application

A Dissertation Submitted to the Graduate School in Partial Fulfillment of the Requirement for the Doctor Degree of Philosophy

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摘 要

随着电子信息技术的飞速发展，电子元器件的工作频率和集成度不断提高，作为电子元器件关键组成部分之一的磁性元器件必然要向微型化、集成化、高频化方向发展。但是传统的铁氧体材料饱和磁化强度较低，受自身性质的限制不能在 GHz 频率范围具有较高的磁导率，因此不能满足当前电磁元件的需求。目前，具有高饱和磁化强度的软磁薄膜越来越受到重视，成为高频磁性材料研究的热点。为了使软磁薄膜能够具有好的高频特性，它不仅需要具有高的饱和磁化强度和高磁导率，而且需要具有较大的电阻率以及适当大的面内磁各向异性场，以抑制涡流损耗和提高自然共振频率。尽管目前已有许多高频软磁薄膜的研究，但是研究结果表明很难使薄膜同时获得较高的饱和磁化强度、较大的电阻率以及高自然共振频率。因此进一步研究在保持较高饱和磁化强度的情况下提高薄膜的电阻率和高频性能是十分必要的。因为 Fe-Ni 合金具有高饱和磁化强度和较低的矫顽力，可在 GHz 频段获得较高的磁导率而备受关注。因此进一步研究 Fe-Ni 基高频软磁薄膜及其在高频电磁器件中的应用是非常必要的。

本研究的主要研究内容及结果如下：

（1）采用磁控溅射的方法，在无外加诱导磁场的情况下溅射制备了一系列 $Fe_{80}Ni_{20}$-O 纳米晶软磁薄膜。研究发现在沉积薄膜时通入少量的氧气，可以获得具有面内磁各向异性的 $Fe_{80}Ni_{20}$-O 薄膜样品，而且改变通入氧气的量就可以调控薄膜样品的面内磁各向异性场。测试结果还表明增加通入氧气的量可以在保持较大饱和磁化强度的情况下，增加薄膜样品的电阻率。当氧气流量比从 0.75%增加到 3%时，$Fe_{80}Ni_{20}$-O 薄膜样品的自然共振频率从 2.2 GHz 增加到 5.9 GHz，电阻率从 56.7 μΩ cm 增加到 108 μΩ cm。

（2）研究发现单层纳米晶薄膜不能在较大厚度下保持好的软磁性能，因此我们在 $Fe_{80}Ni_{20}$-O 薄膜中加入氧化物相，制备了具有不同中间层厚度的 $[Fe_{80}Ni_{20}\text{-O}/SiO_2]_n$ 纳米多层膜样品。研究结果表明，仅通过改变非磁性中间层的厚度就可以调控多层膜样品的面内磁各向异性场，进而可以大范围地调控多层膜样品的自然共振频率。此外，增加氧化物中间层的厚度可以在保证饱和磁化强度

和磁导率较大的情况下，明显提升多层膜的电阻率。

（3）基于高频性能最优的纳米软磁多层膜样品的制备条件，我们在保持磁性层和氧化物中间层厚度不变的情况下制备了总厚度不同的$[Fe_{80}Ni_{20}\text{-}O/SiO_2]_n$多层膜样品。我们分别研究了这些多层膜样品的微观结构、表面形貌、磁学和电学性能，测试结果表明，多层膜样品的总厚度基本不影响样品优异的磁学性能，这为下一步研究平面电感器件提供了实验基础。

（4）研究探索了平面螺线管电感器件的 MEMS 加工工艺，选择综合性能最优的$[Fe_{80}Ni_{20}\text{-}O/SiO_2]_n$多层膜，制备成平面螺线管电感样品的磁性芯层，并对有无磁性芯层的电感样品进行了性能测试和分析，测试结果表明在平面电感中引入高频软磁薄膜可以明显地提高电感的电感值和品质因数。

关键词：Fe-Ni 基软磁薄膜；高频性能；高频平面电感

Abstract

With the rapid development of electronic information technology, as one of the key part of the electronic device components, the operational frequency and integration degree of the electromagnetic devices must become miniaturized and integrated and desired for high frequency implication. The traditional ferrite magnetic materials with low saturation magnetization cannot have high permeability in high-frequency (GHz range) restricted by their own properties, so they could not meet the needs of current electromagnetic devices. Therefore，the soft magnetic films with high saturation magnetization have attracted more attentions, and became the studying focus of the high frequency magnetic materials． In order to produce soft magnetic film materials with excellent high-frequency, magnetic and electrical properties in GHz range, it would require the soft magnetic film materials to have high saturation magnetization M_s, high permeability, high resistivity and appropriate in-plane uniaxial magnetic anisotropy H_k, so as to effectively suppress eddy current loss and to possess resonance frequency f_r. Although films with a large H_k and a high f_r have been achieved in some previous studies, it has always been very challenging to simultaneously obtain a large M_s, a high resistivity and a high f_r. For this reason, it is highly desirable to find a method to efficiently increase resistivity and adjust the high-frequency magnetic characteristics of soft magnetic films without reducing saturation magnetization. Fe-Ni alloy have excellent soft magnetic performances including high saturation magnetization M_s and low coercivity H_c, so Fe-Ni alloy thin films can obtain high permeability in GHz range. Therefore, it is necessary to investigate Fe-Ni based high-frequency soft magnetic films and the application for high-frequency electromagnetic devices.

The main contents and results of this paper were outlined as following:

(1) The $Fe_{80}Ni_{20}$-O nanocrystalline soft magnetic alloy films were fabricated by magnetron sputtering without applying inducing magnetic field. The results showed

that adding a very low dose of oxygen into $Fe_{80}Ni_{20}$ alloy films during deposition, the $Fe_{80}Ni_{20}$-O alloy films with in-plane magnetic anisotropy can be achieved. And the adjustable magnetic anisotropy fields can be obtained by changing the amount of oxygen introduced during the film formation. Moreover, by increasing the oxygen concentration, a higher resistivity can be achieved in the film, while its saturation magnetization can remain at a large value. By increasing the oxygen flow ratio from 0.75% to 3%, $Fe_{80}Ni_{20}$-O alloy films could be achieved with an adjustable resonance frequency f_r from 2.2 to 5.9 GHz, and a high resistivity from 56.7 to 108 μΩ cm.

(2) The single-layer nanocrystalline films cannot obtain preferable magnetic properties at the thicker thickness. So we fabricated $[Fe_{80}Ni_{20}\text{-O}/SiO_2]_n$ multilayer thin films by adding oxide phases into $Fe_{80}Ni_{20}$-O alloy films, and demonstrated that the in-plane magnetic anisotropy fields are only dependent on the thickness of the nonmagnetic insulated SiO_2 interlayer, and then adjustable resonance frequency can be obtained in a broad range. Moreover, increasing the thickness of SiO_2 interlayer, the higher resistivity can be achieved while the saturation magnetization and permeability are still relatively large.

(3) Based on the magnetic multilayer films with optimal high-frequency magnetic performance, we maintained the thickness of $Fe_{80}Ni_{20}$-O single layer and SiO_2 interlayer, and fabricated $[Fe_{80}Ni_{20}\text{-O}/SiO_2]_n$ multilayer thin films with different total thickness. We investigated the structural characteristics, surface morphology, electrical and magnetic properties of these multilayer films, respectively. The resulted showed that the multilayer films with different total thickness could keep the same excellent magnetic performances. The thicker $[Fe_{80}Ni_{20}\text{-O}/SiO_2]_n$ multilayer thin films can be used in the research of the planar inductors.

(4) The research explored the MEMS processing technology of the planar solenoid-type inductors, and prepared planar solenoid-type inductors with magnetic core layer using the $[Fe_{80}Ni_{20}\text{-O}/SiO_2]_n$ multilayer thin films with optimum high-frequency magnetic properties. The results from measurements and analyses of the planar solenoid-type inductor samples with or without magnetic core layer indicated that the inductance and quality factor can be significantly improved by