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Analysis of structural, mechanical, and magnetic properties of electroplated NiAg thin films synthesized at different deposition time

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By changing the electroplating deposition duration, such as 15, 30, 45, and 60 minutes, the NiAg thin films have been synthesised by electrodeposition on copper substrate at a uniform current density of 1 A/dm^2. The NiAg thin films were adhered to the substrate at a consistent electrolytic solution temperature of 30°C, and the created electrolyte's pH was maintained within a range of 7 to 8. EDAX, XRD, and SEM techniques have all been used to investigate the structural investigation of electrodeposited NiAg thin films. The FCC crystal structure can be seen by the X-ray diffraction analysis pattern, which indicates that the average crystalline size of the NiAg-coated thin films was between 31 and 64 nm.Images of coated thin films taken with a scanning electron microscope show that, even with longer deposition times, the surface morphology of electrodeposited NiAg thin films is consistent. The Vibrating Sample Magnetometer (VSM) examination was used to investigate the magnetic characteristics. With a greater saturation magnetization of 7.6930 × E^{-3} emu/cm^3 and a lower coercivity of 92 Oe, the NiAg film deposited at 15 minutes shows a better soft magnetic nature. According to the electrochemical analyses of plated NiAg thin films, the NiAg film coated at the 60 -minute deposition period showed the lowest corrosion rate, measuring 0.047286 mm/year with a polarization resistance of 3900.9 Ω. NiAg thin films' mechanical and magnetic characteristics make them suitable to produce MEMS and NEMS based devices.

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Electrochemical studiesand corrosion rate

1. Introduction

Nickel-silver (NiAg) thin films are widely used in various technological applications such as electronic devices, sensors, and catalysis due to their desirable properties including high conductivity, corrosion resistance, and mechanical strength. Electrodeposition is a commonly employed technique for fabricating NiAg thin films owing to its simplicity, cost-effectiveness, and ability to control film composition and morphology [1-3]. In this literature review, we summarize recent advancements in the electrodeposition of NiAg thin films, focusing on the methods employed, key findings, and potential applications.

Nickel-silver (NiAg) thin films represent a significant class of materials with versatile properties, making them indispensable in various technological domains. These thin films are predominantly fabricated using electrodeposition techniques owing to their ease of fabrication, cost-effectiveness, and precise control over film properties. In this detailed introduction, we delve

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into the significance of NiAg thin films, their fabrication through electrodeposition, and their relevance in contemporary technological applications.

Significance of NiAg Thin Films: NiAg thin films combine the advantageous properties of both nickel (Ni) and silver (Ag), offering a unique blend of characteristics that are highly desirable across multiple industries. Nickel contributes to the films' corrosion resistance, mechanical durability, and magnetic properties, while silver enhances their electrical conductivity and catalytic activity [4-9]. This synergistic combination makes NiAg thin films suitable for a broad spectrum of applications, including microelectronics, sensors, catalysis, and flexible electronics.

Fabrication through Electrodeposition: Electrodeposition stands out as a preferred method for synthesizing NiAg thin films due to its versatility, scalability, and ability to precisely control film composition and morphology. During electrodeposition, a direct current (DC) or pulse current is applied between two electrodes immersed in an electrolyte solution containing nickel and silver ions. The ions migrate towards the cathode (substrate), where they undergo reduction and deposit as a thin film. By adjusting parameters such as electrolyte composition, pH, temperature, and current density, researchers can tailor the properties of the deposited NiAg films to meet specific application requirements.Relevance in Technological Applications: The unique properties of NiAg thin films make them indispensable across various technological domains [10 – 12].

In microelectronics, NiAg films serve as vital components in interconnects, contacts, and metallization layers, facilitating efficient electrical conductivity and reliability in integrated circuits and electronic devices. Moreover, their corrosion resistance and catalytic activity render them suitable for sensor electrodes, where they enable robust sensing capabilities in environmental monitoring, biomedical diagnostics, and industrial process control [13 -14]. Additionally, NiAg thin films deposited on flexible substrates hold promise for emerging applications in flexible electronics and wearable devices, offering mechanical flexibility, durability, and electrical performance in conformal and stretchable form factors.

NiAg thin films synthesized through electrodeposition represent a crucial class of materials with diverse properties and applications. The facile fabrication process, coupled with precise control over film properties, makes electrodeposition an attractive technique for producing NiAg thin films tailored to specific industrial and technological needs. Moving forward, further research and development efforts are warranted to explore novel applications, optimize fabrication processes, and enhance the performance of NiAg thin films in existing and emerging technologies.

Electrodeposition of NiAg thin films can be achieved through various methods including direct current (DC) electrodeposition, pulse electrodeposition, and electroless deposition. DC electrodeposition involves applying a constant current or voltage between two electrodes immersed in an electrolyte solution containing Ni and Ag ions, resulting in the deposition of NiAg thin films on the substrate. Pulse electrodeposition utilizes periodic variations in the applied potential or current to control the deposition process, offering improved film quality and composition control compared to DC electrodeposition [15 -17]. Electroless deposition, on the other hand, involves the chemical reduction of metal ions on the substrate surface without the need for an external power source, enabling deposition on non-conductive substrates.

Several studies have investigated the influence of deposition parameters such as electrolyte composition, pH, temperature, and current density on the morphology, composition, and properties of NiAg thin films. It has been observed that the Ni/Ag ratio in the deposited films can be controlled by adjusting the relative concentrations of Ni and Ag ions in the electrolyte solution. Additionally, variations in deposition parameters have been shown to affect the crystal structure, grain size, and surface roughness of the deposited films, ultimately influencing their electrical, mechanical, and corrosion resistance properties.

NiAg thin films synthesized via electrodeposition have found applications in diverse fields including microelectronics, MEMS (Micro-Electro-Mechanical Systems), sensors, and catalysis [18-19]. Their high conductivity makes them suitable for interconnects and contact materials in electronic devices, while their corrosion resistance and catalytic activity enable their use in sensor electrodes and catalytic converters. Furthermore, NiAg thin films deposited on flexible substrates hold promise for flexible electronics and wearable devices due to their mechanical flexibility and adhesion properties. Electrodeposition offers a versatile and efficient method for synthesizing NiAg thin films with tailored properties for various applications. Further research is needed to optimize deposition parameters, improve film quality, and explore new applications of NiAg thin films in emerging technologies.

2. Experimental part

NiAg thin films were deposited on the copper substrate with suitable high-grade chemicals by varying the deposition time such as 15, 30, 45, and 60 minutes at bath temperature of 30°C. The chemical composition of electrolytic bath for NiAg thin film and the other bath details are shown in the table 1. In order to coat the high quality NiAg thin films, copper plate and stainless-steel plate have been used as cathode and anode with optimized size of length 7 cm & breath of 1.5 cm. Substrate cleaning process plays a dominant role to decide the quality of the electroplated thin films. Initially both the substrates were cleaned by high grade emery sheet followed by soap solution and final dilute sulfuric acid cleaning over the period of 2 min. All the substrates are rinsed with triple distilled water just before the deposition. All the surface area of cathode was masked off except the desired area for NiFeAg coatings using adhesion tape. All the desired chemical reagents have been dissolved in triple distilled water to make electrolyte bath.

Table 1. Experimental data for the NiAg films.

S. No	Name of the Parameters	Data (g/l)
1	Nickel Sulphate	46.41
2	Tri Sodium Citrate	180.6
3	Silver Nitrate	10
4	pH Value	7
5	Temperature	30°C
6	Current density	1 A/dm^2
7	Deposition Time	15,30, 45 and 60 minutes

The pH of the prepared bath was modified in between 7 – 8 by adding a few drops of ammonia solution. NiAg thin films were synthesized by applying a current of 60 mA/cm^2 through the electrodes for the duration of 15, 30, 45 and 60 minutes at constant temperature of 30°C. All the synthesized NiAg thin films are dried for few minutes in open atmosphere and are packed using air lock covers for various characterization analysis like SEM – EDAX, XRD and VSM etc.,

3. Results and discussion

3.1. Chemical composition analysis

Each of the NiAg film that was electroplated at a distinct deposition time was subjected to structural characterization examination using tools like SEM and EDS. The resulting EDS spectrum of coated NiAg films is depicted in figure 1, and table 2 provides the corresponding chemical composition information. Together with the base copper peak, all the synthesized films showed prominent peaks indicating the presence of Ni and Ag.

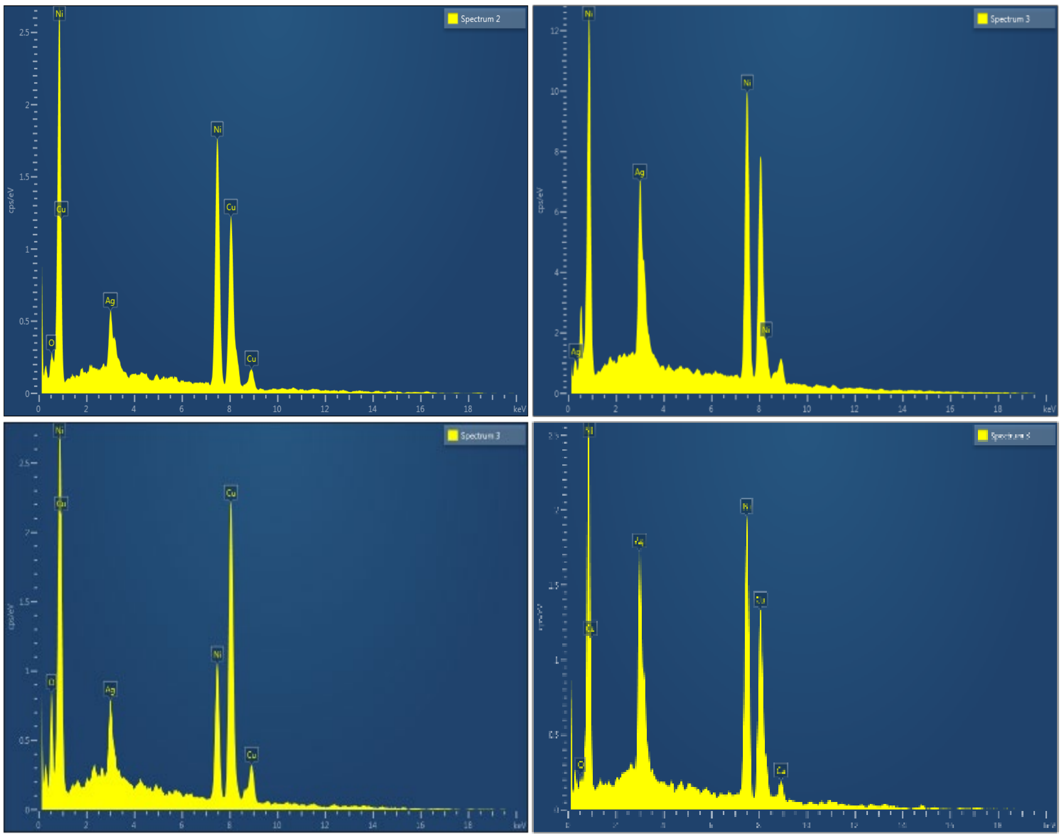


Fig. 1. EDS spectrum of NiAg thin films (A): Coated at 15 minutes (B):Coated at 30 minutes (C): Coated at 45 minutes (D): Coated at 60 minutes.

Table 2. EDS data for NiAg films at different deposition time.

Sample Description	Element	Line Type	Wt%	Atomic %
NiAg – 15 min	Ni	K series	84.92	87.5
	Ag	L series	10.08	12.5
NiAg – 30 min	Ni	K series	83.24	81.99
	Ag	L series	16.76	18.01
NiAg – 45 min	Ni	K series	80.72	67.96
	Ag	L series	19.28	32.09
NiAg – 60 min	Ni	K series	76.86	82.07
	Ag	L series	23.14	17.93

The EDS data made it abundantly evident that when the deposition time got higher, the presence of Ni content steadily dropped, and the Ag content grew. With a weight percentage of 76.86 and an Ag content of 23.14, the NiAg film coated after 60 minutes of deposition had the highest level of Ag.

More silver ions have the chance to diffuse to the substrate's surface and take part in the reduction reaction as the deposition time increases, which results in the creation of silver atoms in the thin layer.More effective reduction of silver ions at the cathode (substrate surface) is possible with longer deposition durations. The amount of silver in the deposited thin film rises because of a larger percentage of silver ions being reduced to generate silver atoms. The distribution of current across the substrate surface has an impact on electroplating. A higher silver content across the thin film and more uniform deposition can result from longer deposition times as the current distribution becomes more uniform. Overall, several electroplating process-related variables, such as diffusion, kinetics, electrodeposition rate, competing reactions, and current distribution, contribute to the rise in silver content in NiAg electroplated thin films with longer deposition durations.

3.2. SEM analysis of NiAg thin films

Using techniques like SEM, structural characterization analysis was performed on every NiAg film that was electroplated at a different deposition time. Figure 2 shows the resultant SEM micrographs of coated NiAg film. The synthesized NiAg films have bright, crack free surface morphologies with spherical shape structures. The Ni and Ag particles start to agglomerate and leads to transform rod shape structure from spherical shape while increasing deposition time (15 to 60 minutes).A relatively sparse and irregular morphology with small particle size may be seen in SEM pictures of NiAg thin layers coated at 15 minutes time.

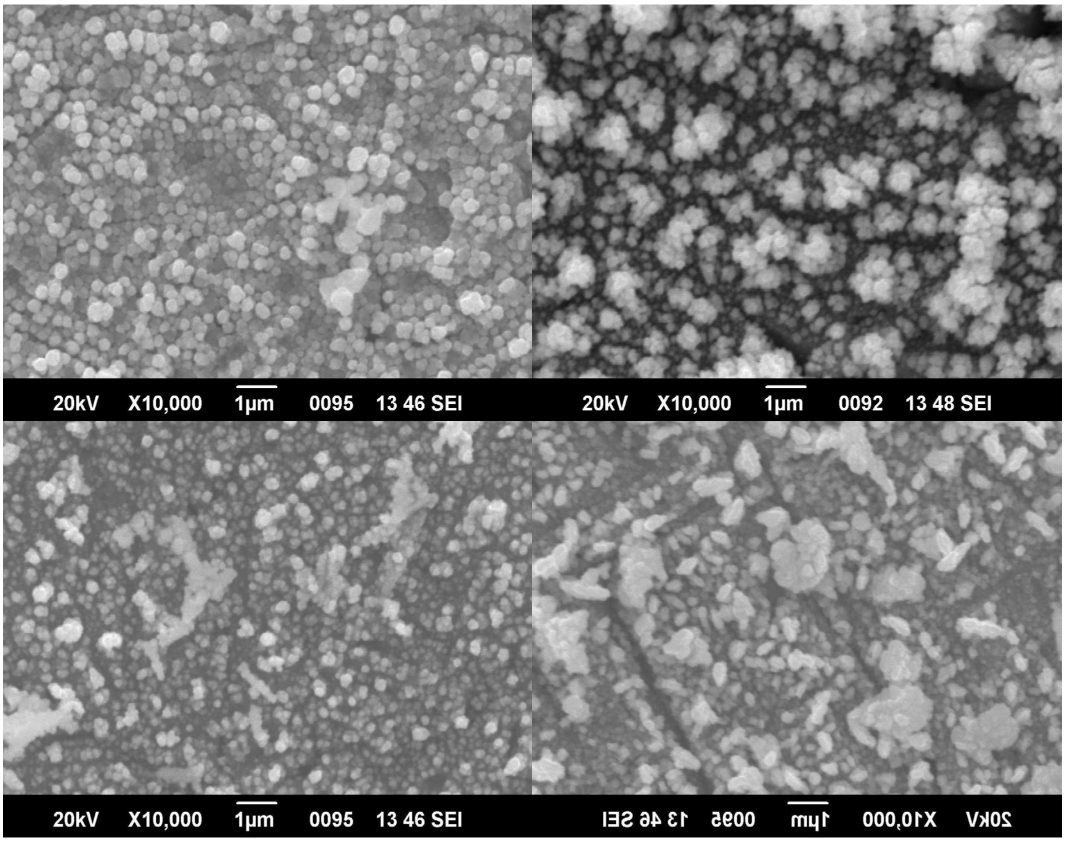


Fig. 2. SEM micrographs of NiAg thin films (A): Coated at 15 minutes (B): Coated at 30 minutes (C): Coated at 45 minutes (D): Coated at 60 minutes.

There may be some indication of nucleation sites and early grain development on a somewhat rough surface. Grain boundaries could seem less distinct, suggesting insufficient silver assimilation and partial coverage. Grain size and surface coverage may have increased in SEM pictures as compared to the 15-minute deposition. Grain boundaries might become sharper, indicating better crystalline development and silver inclusion.

Surface homogeneity and grain size may show a considerable rise in SEM pictures for the film coated at 60 minutes duration. There may be fewer flaws in the film, giving the impression of density and homogeneity. The creation of a continuous NiAg alloy film with a higher silver content (also confirmed with EDS data) is indicated by well-defined grain boundaries. Surface roughness may continue to decline, a sign of improved structural integrity and film quality.

3.3. XRD results for NiAg thin layers

XRD was used to examine the structural characteristics of every NiAg film that was electroplated at a different deposition time. Figure 3 shows the coated NiAg film XRD results, and Table 3 gives the related XRD data. The coated NiAg films have a crystalline nature with a crystalline size of a few tens of nanometers, as demonstrated by the XRD data.

When the results of the NiAg thin films were compared to the standard JCPDS data (JCPDS card No. 04-0783 and No. 04-0850), it was discovered that the Ag(111), Ni(111), and Ni(200) crystal planes of the NiAg thin layers displayed the FCC structure.

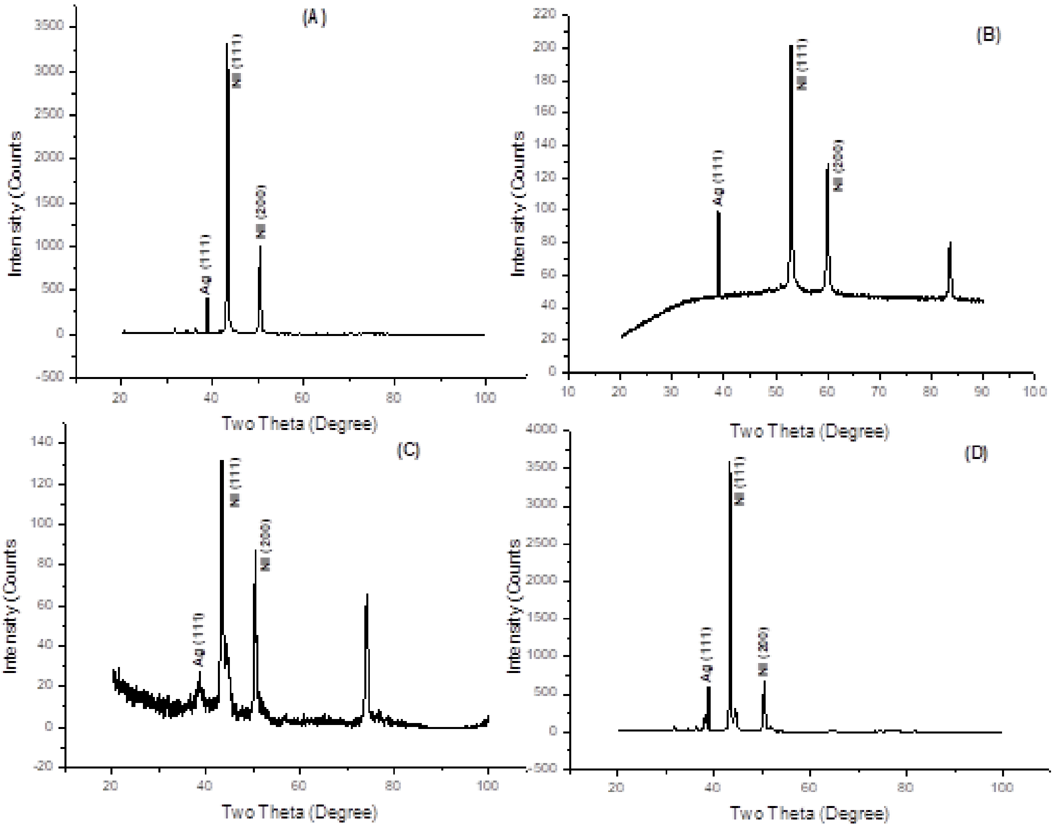


Fig. 3. XRD graphs of NiAg thin films (A): Coated at 15 minutes (B): Coated at 30 minutes (C): Coated at 45 minutes (D): Coated at 60 minutes.

The NiAg film synthesized at 60 minutes duration exhibited the crystalline size of 31.72 nm which is significantly high value compared with the NiAg film coated at 15 minutes duration (17.68 nm). The XRD results show that the crystalline size has been increased upon increasing the electrodeposition time.

Table 3. XRD data for NiAg films at different deposition time.

Sample details	**Two Theta (degree)**	**FWHM**	**d value Å**	**Crystalline size (D) (nm)**	**Stain (10^{-4})**	**Dislocation density ($10^{15}/m^2$)**
NiAg – 15 min	43.325 °	0.462	2.08676	17.68	18.71	3.20
NiAg – 30 min	43.279 °	0.322	2.08888	25.35	13.07	1.56
NiAg – 45 min	43.303 °	0.308	2.08775	26.52	12.50	1.42
NiAg – 60 min	42.936 °	0.257	2.10475	31.72	10.43	0.99

Larger Ni and Ag crystallites can form because of longer deposition durations because they increase atom mobility on the surface and allow atoms to locate energetically favorable locations. The coalescence of Ni and Ag small grains into larger ones is facilitated by a rise in the diffusion length of adatoms, also known as adsorbed atoms, with time. Longer deposition durations may also have an annealing effect, in which the development of larger, more perfect crystals is encouraged by heat processes that reduce flaws and reorganize the crystal structure. Smaller grains begin to converge and merge as they get bigger, creating larger crystalline structures. A longer deposition period causes this grain coalescence to become more noticeable, which increases the crystalline size overall.

3.4. VSM results of NiAg

VSM was used to examine the magnetic characteristics of all the NiAg film that was electroplated at a different deposition time. Figure 4 shows the VSM results of coated NiAg film, and Table 4 gives the related VSM data. The magnetic characteristics of NiAg (Nickel-Silver) thin films are largely dependent on the electrodeposition time. The electrodeposition time can have an impact on a number of aspects that affect the magnetic characteristics of NiAg thin films, including composition, crystal structure, grain size, and microstructure.

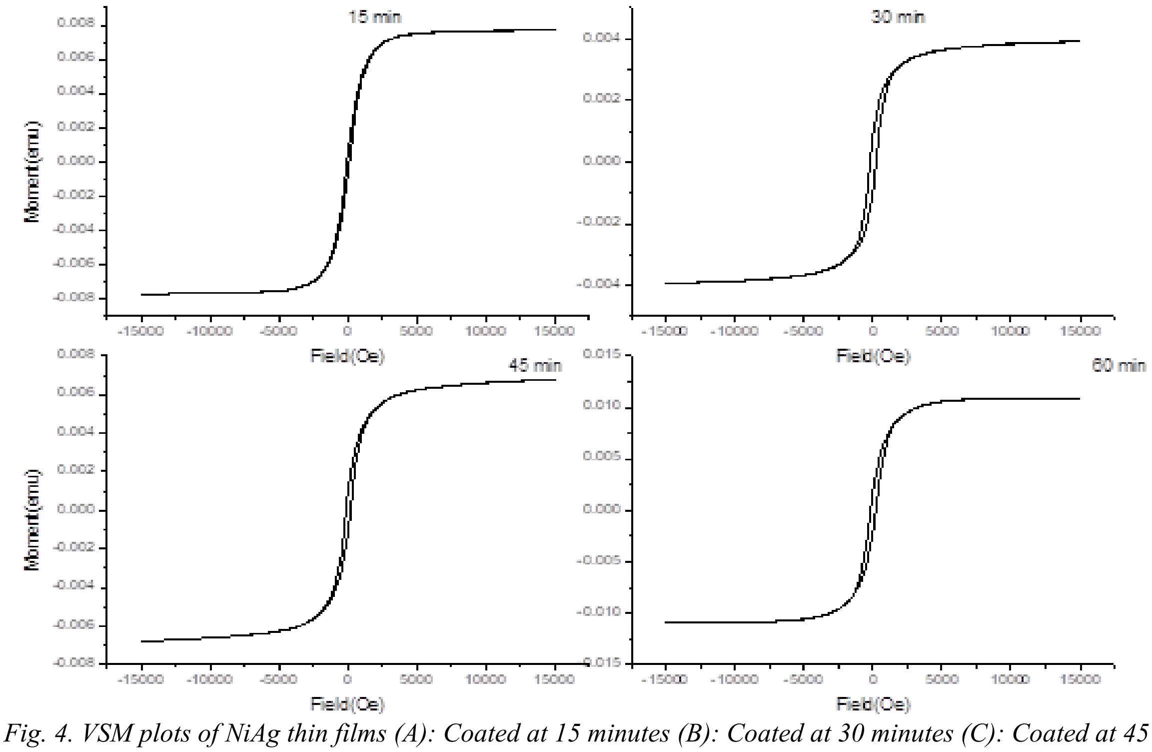


Fig. 4. VSM plots of NiAg thin films (A): Coated at 15 minutes (B): Coated at 30 minutes (C): Coated at 45 minutes (D): Coated at 60 minutes.

The length of the deposition process during electrodeposition directly affects the NiAg thin film's growth kinetics. Larger grain sizes and alterations in crystal structure are usually the outcome of longer deposition durations. Longer deposition durations, for example, can result in the development of bigger crystallites with preferred crystallographic orientations, which could affect the thin film's domain structure and magnetic anisotropy.

By affecting the relative deposition rates of nickel and silver, electrodeposition time influences the composition of NiAg thin films. Longer electrodeposition periods have the potential to cause one metal to be preferentially deposited over another, changing the thin film's overall composition.

Table 4. VSM data for NiAg films at different deposition time.

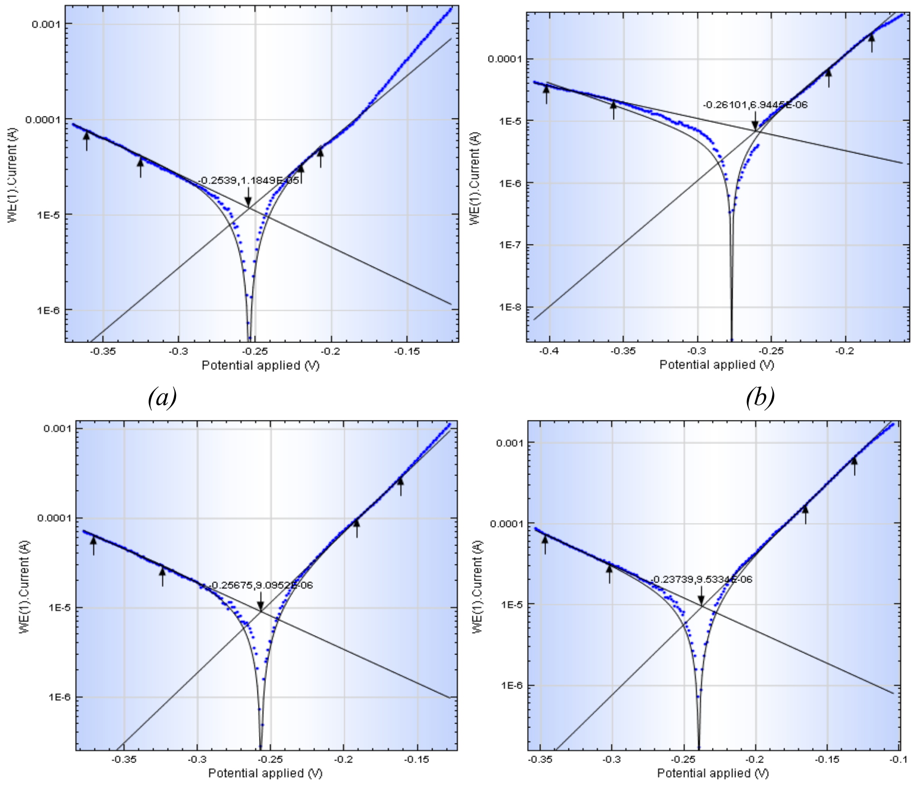
Sample details	**Magnetization (Ms) (× 10^{-3}) emu**	**Retentivity (Mr) (× 10^{-6}) emu**	**Mass gram**	**Coercivity (Oe)**	**Squareness S**
NiAg – 15 min	7.6930	647.29	0.19300	92	0.0841
NiAg – 30 min	6.7875	1180	0.17000	167	0.1738
NiAg – 45 min	10.913	1978.4	0.18100	183	0.1813
NiAg – 60 min	3.9231	956.48	0.18100	223	0.2438

The electrodeposition time can influence the magnetic anisotropy of NiAg thin films, which refers to the direction-dependent magnetic properties. The NiAg film coated at 15 minutes time has the lowest coercivity of 92 Oe as compared with the film coated at 30,45, and 60 minutes. The coercivity value has increased while increasing the deposition time and the film coated at 60 minutes has thelarge value (223 Oe) of coercivity with low magnetization value of 3.9231 (× 10^{-3}) emu. This may be due to the Changes in grain size, crystal structure, and composition of NiAg thin layers at 60 minutes of deposition time which also affects the magnetic anisotropy energy, leading to modifications in the preferred magnetization direction and magnetic domain configuration.

Longer deposition times for NiAg may lead to higher coercivity (also confirmed with VSM results) due to the formation of larger and more well-defined magnetic domains within the NiAg thin film. The dynamics of magnetization processes in NiAg thin films, including magnetization reversal and domain wall motion, mobility of magnetic domain walls and the relaxation dynamics of magnetization can also be influenced by the variations in electrodeposition time, thereby impacting the overall magnetic behaviour of the NiAg thin film.

3.5. Electrochemical analysis of NiAg thin films

Electro chemical analysis was used to examine the corrosion behaviors of all the NiAg film that was electroplated at a different deposition time.



(a) *(b)*

(c) *(d)*

Fig.5. Tafel plots of NiAg thin films (A): Coated at 15 minutes (B): Coated at 30 minutes (C): Coated at 45 minutes (D): Coated at 60 minutes.

Figures 5 & 6 shows the electro chemical results of coated NiAg film, and table 5 gives the related corrosion data.The electrochemical response yielded the Nyquist and Tafel graphs. Based on the EIS measurements, the charge transfer resistant (Rct) values were computed.

To get the corrosion current densities, Tafel polarization analysis was carried out by extending the cathodic and anodic curve to the applied potential. The Tafel plots were used to derive the corrosion potential (Ecorr), corrosion current density (Icorr), and polarization resistance (Rp). From the Tafel plots, the cathodic and anodic slopes of βa and βc were also noted.

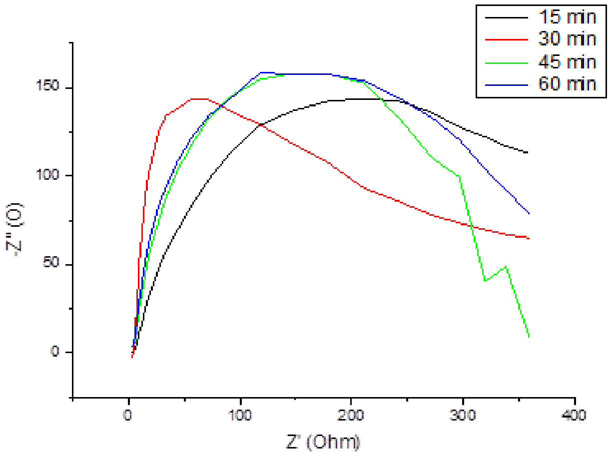


Fig. 6. Nyquist plots of NiAg thin films (A): Coated at 15 minutes (B): Coated at 30 minutes (C): Coated at 45 minutes (D): Coated at 60 minutes.

Table 5. EIS (Polarization) data for NiAg films at different deposition time.

Sample details	**ba (V/dec)**	**bc (V/dec)**	**Ecorr, Obs (V)**	**jcorr (A/cm²)**	**icorr (A)**	**Corrosion rate (mm/year)**	**Polarization resistance (Ω)**
NiAg – 15 min	0.15009	0.076558	-0.25419	1.47E-05	1.47E-05	0.17105	1495.8
NiAg – 30 min	0.12007	0.059117	-0.23992	9.63E-06	9.63E-06	0.11186	1787.1
NiAg – 45 min	0.14814	0.067199	-0.25687	1.09E-05	1.09E-05	0.12673	1840.9
NiAg – 60 min	0.12215	0.05216	-0.27724	4.07E-06	4.07E-06	0.047286	3900.9

The NiAg film coated at 15 minutes time has the lowest polarization resistance of 1495.8 Ω as compared with the film coated at 30,45, and 60 minutes. The polarization resistance value has increased while increasing the deposition time and the film coated at 60 minutes has the large value (3900.0 Ω) of polarization resistance with low corrosion rate value of 0.047286 mm/year. The microstructure and grain size of coated NiAg thin films are directly influenced by the electrodeposition time. Extended deposition durations usually produce microstructures with more compact grain sizes.

Because reduced grain boundary density might prevent corrosive elements from diffusing into the film, coarser grain structures with extended deposition durations may show improved corrosion resistance. This may also due to the surface morphology, which includes surface porosity and roughness, can vary with electrodeposition time and has a substantial impact on the corrosion behaviour of NiAg thin films.

Extended periods of deposition could lead to denser and smoother film surfaces, which would improve the film's ability to withstand corrosion by limiting the flow of corrosive chemicals to the substrate underneath. Longer deposition times may encourage the development of shielding surface layers of NiAg over the substrate that act as barriers against corrosive species, improving the film's resistance to corrosion.The Nyquist plot's semicircle demonstrates how the charge transfer reaction that governs the corrosion process is caused by the accumulation of NiAg on the

Cu plate. Diameter of semicircle indicates how much electrons are transferred over the metal surface in an exposed area, and it is inversely correlated with corrosion rate.

3.6. Mechanical testing of NiAg thin films

The Vickers Hardness method was used to examine the mechanical characteristics of all the NiAg film that was electroplated at a different deposition time. Table 6 shows the VHN and thickness value of coated NiAg film. The thickness of the coated NiAg varies from 13 to 20 microns and the film coated at 60 minutes duration has the highest hardness number of 185 VHN compared with other NiAg films. Using bend and scratch test, the adhesion level of NiAg films have been analyzed and found that the NiAg layers have strong adhesion with substrate.

Table 6. Thickness and hardness data for NiAg films at different deposition time.

S. No	Sample details	Coating thickness (Microns)	Hardness value (VHN)
1	NiAg – 15 min	13	85
2	NiAg – 30 min	14	122
3	NiAg – 45 min	16	135
4	NiAg – 60 min	20	185

4. Conclusion

The NiAg thin coatings were successfully synthesized by varying the electrodeposition time at constant current density. The structural analysis performed using XRD showed a single FCC structural growth in the samples, which explains the stability of the synthesized coating. The crystalline size and hardness of the NiAg samples prepared at 60 minutes was larger than that of NiAg samples prepared at 15, 30 and 45 minutes of duration, which is also evident in the SEM image, the increase in crystalline size with increased deposition time during the electrodeposition of NiAg thin films can be attributed to enhanced nucleation and growth dynamics, greater atom mobility, reduced defect density, and grain coalescence. The corrosion rate of the NiAg sample coated at 60 minutes was very low as compared with the other NiAg films. All the coated NiAg thin layers exhibited the ferromagnetic nature and the coercivity value was increased while increasing with deposition time. On the whole, the crystalline size, hardness, corrosion resistance and coercivity of the coated NiAg films were increased while increasing with deposition time from 15 to 60 minutes. This may be due to the Changes in grain size, crystal structure, and composition of NiAg thin layers at 60 minutes of deposition time.

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