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Design and Synthesis of Neutral Density Filter in Visible Range for Color Sorting Applications

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This paper provides the design, construction, and characterization of a neutral density filter (NDF) for the visible spectrum and investigate the thickness effect on their various properties. In this work, Nichrome single layer was designed by the Essential Macleod modelling software. The Co-evaporation ofss nickel and chromium was used to construct a filter on a glass substrate by thermal vacuum coating unit under high vacuum. Low thickness supports amorphous nature examined by XRD diffractogram. Energy-Dispersive X-ray Spectroscopy (EDAX) examinations of the thin films revealed that high thickness has slight variation in constant stoichiometry for the chosen Nickel/Chromium ratio (80:20) during co-evaporation and confirm the deposition is successfully done. Additionally, Atomic Force Microscopy (AFM) analysis revealed that surface roughness decreased from 6.034 to 2.895 in correlation with increased layer thickness. These methods made it possible to produce NDFs with optical densities ranging from 0.5 OD at 10 nm film thickness to 2.7 OD at 70 nm thickness. As a result, for steady and long-lasting NDF, the spectral fluctuation of thin filters in our case is lowest at 10 nm (ΔOD= 0.08). A good match data was found between optical transmission and absorption spectra of the designed and the deposited films. The designed NDF's applicability and effectiveness for color sorting tasks were subsequently examined, marking a promising step towards advancing optical engineering solutions in this specific application domain.

Keywords: Attenuator; Optical coating; Absorption; Thin film; Transmission

1 Introduction

The protection of human eyes, skin, and laser detectors in high intensity laser systems needed different optical technologies[1-3]. The optical filter is used to modify the spectrum characteristics and lower the power intensity in order to provide safe and comfortable working circumstances[4-5]. Neutral density filter (NDF) is a filter that is used to evenly attenuate incident light, independent of wavelength[6] named as an attenuator but all attenuator is not neutral. Neutral density filters play a vital role in many applications such as spectrophotometers, photography, instrumental calibration, and chemical detection[7]. Neutral density filters are designed to reduce transmission by absorption or reflection across a portion of a specific spectrum[8]. Neutral Density Filter are typically delimited by their optical density (OD) which measures the amount of intensity blocked by the filter. If the films are absorptive and reflective also then:

$R+A+T=1$

To calculate the OD is given by[6]

$OD = -Log\, T$

Where $T=I_O/I_T$

Wherein: I_O = incident light intensity

I_T = transmitted light intensity

NDF can be produced by glass filter formed of glass to which various inorganic and organic compounds have been added. To reduce the cost of ND filters glass filters are widely used which are simply based on absorption phenomenon. However, glass ND filters don't have consistent spectral properties in the visible spectrum hence their use for low optical density is discarded[9], due to absorptive nature heat built up and possibility of crack formation is increased[10] and hence thin film based NDF are preferred. Thin film based NDF have less ripple than absorptive filters based on iron and cobalt oxides dissolved in the glass. Optical coating includes a thin layer of metal and metal alloy which is deposited on substrate by PVD techniques. For deposition of hard coating, several PVD techniques are employed such as Thermal Evaporation[11], Sputtering[12-15], E-beam and arc processes[16-17]. Metal or Metal alloy thin film

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absorb a portion of incident light, the amount of light absorbed being a function of thickness of film[18]. Condition for designing NDF for a particular application are that substrate should be transparent and materials should be isotropic, have no inter band transition in a specific region and less electron mean free path[19]. Here we are designing a ND filter for color sorting application which required precise control of light reaching to the camera to avoid the saturation. To make ND Filters, metals can be coating materials but their film with less than 5 nm are not easy to control[20]. So, a design which have film thickness more than 5 nm is preferred for metal films. This can be avoided by using multilayer coating which is again a tedious job increasing the cost of filters. In present case widely acceptable material Nichrome alloy is used as a single layer of coating which is easy to fabricate, cheap and have good stability and reliability. These characteristics render them appealing for many industrial and optical applications[21-23]. Because of its excellent thermal stability, low temperature coefficient of resistance (TCR), and high resistivity, several researchers have focused on its electrical characteristics in the literature, particularly as a thin film resistor. For example, the structural, electrical, and mechanical properties of Nichrome thin films deposited employing sputtering method units were described by Chuang *et al.*[24], Petrovic *et al.*[25], Ajay *et al.*[26], Wu *et al.*[27], Vijay *et al.*[28], and Zhou *et al.*[29]. However, nichrome alloy is perfect for creating ND filters because it has some unique optical properties, such as the ability to filter throughout a visible range of wavelengths and exhibit good neutrality over a visible spectrum[30]. Banning[31] using chromel having densities in ranging 2 and Okami *et al.*[9] have transmission variation is 10% using multilayer structure which is costly to fabricate. In our work, we concentrated on the co-evaporation method to maintain the constant stoichiometry after the deposition which is not covered in literature part. Two boats were used separately having Ni and Cr to vary their index in thermal evaporation technique because mixture of Ni and Cr is difficult to control during evaporation in single boat due to different vapor pressure of both[32]. The visible range 400 nm to 700 nm is considered for design optimization. A variation in thickness controls the OD of filter from 0.5 to 2.7 at reference wavelength of 520 nm. In my work the range of optical density lies in between 0.1 to 3. The optimal values of optical density for spectrophotometer are in range from 0.3 to 0.9[33] and my values are closely related to this. The optical densities of 3.0 or less are ideal for color sorting and separating spectral orders[34].

2 Experimental Details

Nichrome thin films were deposited onto Borosilicate glass substrates using the thermal evaporation technique. The desired film thicknesses of 10, 30, and 70 nm were achieved based on simulation results obtained from Essential Macleod software. Nickel and chromium powder were utilized as target materials, each placed in separate tungsten boats. Borosilicate glass slides measuring 75×25 mm with a thickness of 1.35±5 mm was employed as substrates. Prior to deposition, the glass substrates underwent cleaning with acetone via ultrasonic treatment, followed by rinsing with distilled water and drying using a nitrogen blower. The distance between the tungsten boats and the substrate was maintained at 10 cm. The entire deposition process was conducted within a vacuum chamber under a vacuum pressure of 5×10^{-5} mbar, with a total deposition time of 2 minutes. Film thickness was monitored using in-situ quartz thickness monitor. Optical and structural characterization of the deposited films was performed using a Varian UV-Vis-NIR Spectrophotometer (Cary-5000) and Rigaku X-ray diffraction (Smart lab 3KW), respectively. Surface morphology analysis was conducted using non-contact mode Atomic Force Microscopy (Park NX10), and elemental composition was verified using FESEM-EDS (7610 F Plus/JEOL).

3 Results and Discussion

3.1 Structural Properties

X-ray diffraction (XRD) analysis was conducted on prepared thin film samples using a Rigaku Smart lab 3kw Diffractometer with Cu Kα radiation at room temperature, covering a 2θ range of 20º-80º at a scanning rate of 2º/min. The X-ray diffractogram of the Nichrome thin film is depicted in Fig. 1. For the 10 nm thickness film, no peaks were detected, indicating an amorphous nature with a broad hump near 25°[35-36]. However, at thicknesses of 30 nm and 70 nm, peaks appeared at 39.3° and 44.2°, suggesting crystalline properties of the Nichrome thin films. These peaks correspond to the (200) and (210) planes of Nichrome, exhibiting a cubic structure with space group Pm-3n (223), consistent with JCPDS card number (00-026-0429). The inter-atomic spacing (d) was measured to be 2.044 Å, and the lattice parameter (a=b=c) was determined to be 4.5 Å. No significant peaks associated with impurities or oxide compounds were perceived, confirming the purity of the Nichrome component in the coating.The summarized results are presented in Table 1.

Crystallite size of the sample is premeditated by the Debye Scherrer formula[37].

Crystallite Size $$D = \frac{0.9\,\lambda}{\beta cos\theta} \quad ...(1)$$

In the given context, λ represents the wavelength of the X-rays employed, β denotes the full width at half maximum of the peaks measured in radians, and θ stands for Bragg's angle.

Lattice strain is determined using the following equations.

Lattice strain $$\epsilon = \frac{\beta Cot\theta}{4} \quad ... (2)$$

As we know that non-crystalline films can have excellent optical properties due to their ability to maintain high transparency, tailored refractive indices, controlled absorption characteristics, and smooth surface finishes[38-39]. The amorphous nature of the alloy can be assumed to enhance the corrosion resistance of the materials[40-41]. It has more transmittance having less spectral variation (neutrality) and optical density which is good for specific application such as spectrophotometry, color sorting, photography.

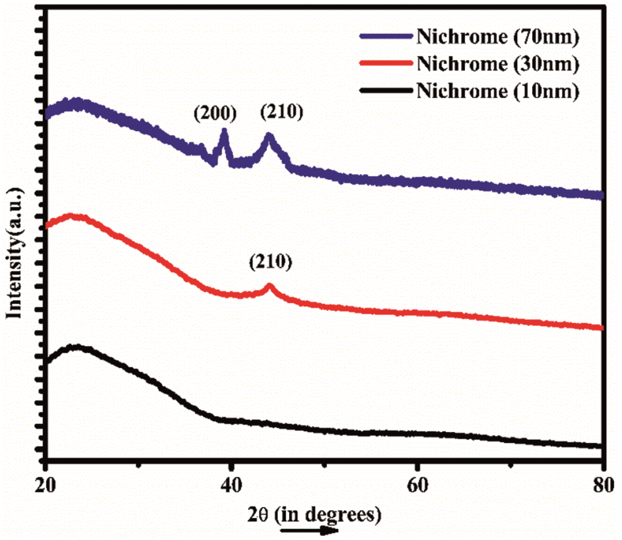


Fig. 1 — XRD diffractogram of Nichrome thin film deposited on glass substrate

A smaller crystallite size of 8.10 nm results peak broadening and lattice strain for thin film having least value of 0.019488 indicating no peak shifting[42].

3.2 Surface Topographical Properties

The 3D topography of Nichrome thin films with varying thicknesses was examined using AFM, covering a scan area of 5µm × 5µm. The topographical analysis indicated uniform coating, suggesting suitability for optical applications. AFM offers insights into the surface roughness of ND filters, crucial for their optical performance. Enhanced smoothness leads to improved light transmission and reduced scattering, enhancing filter neutrality and overall efficiency. Analysis of Fig. 2 reveals that the RMS values of Nichrome thin films indicate high quality, ideal for ND filter applications. Notably, an increase in film thickness corresponds to a decrease in surface roughness and RMS values, declining from 6.034 to 2.895 nm. This reduction is attributed to the absence of columnar structures, a finding supported by SEM analysis. Decreased roughness mitigates scattering losses in ND filters.

The grain size distribution also plays a vital role in roughness AFM used to analyse the grain size and distribution of particles or irregularities in the filter material. A more uniform grain distribution with smaller grains is desirable for better light transmission and filter neutrality. But due to a combined effect of grain growth, self-shadowing and diffusion effect films get smother on increasing the film thickness even after increase of grain size which is effect of deposition method and process parameters taken during deposition[43]. Nichrome 70 nm thickness displays fine grain growth which have less boundaries and smooth surface which means fewer scattering losses in neutral density filter.Smaller grain size distribution increase the neutrality of filter and supportive for attaining high optical density. Good agreement between simulations and actual films has been achieved. This is useful for quality control and optimization of manufacturing processes.

Table1 — Structural parameters of Nichromes thin film along with Average Crystallite size, Lattice strain for numerous thicknesses

Sample	Planes	$2\theta^0$	$d(A^0)$	FWHM(rad.)	Structure	D(nm)	ε
Nichrome (10nm)	-	-	-	-	Amorphous	-	-
Nichrome (30nm)	(210)	44.2	2.044	0.03141593	Cubic crystalline	4.63	0.0193
Nichrome (70nm)	(200) (210)	39.3 44.2	2.290 2.044	0.0138 0.0247	Cubic crystalline	8.10	0.0124

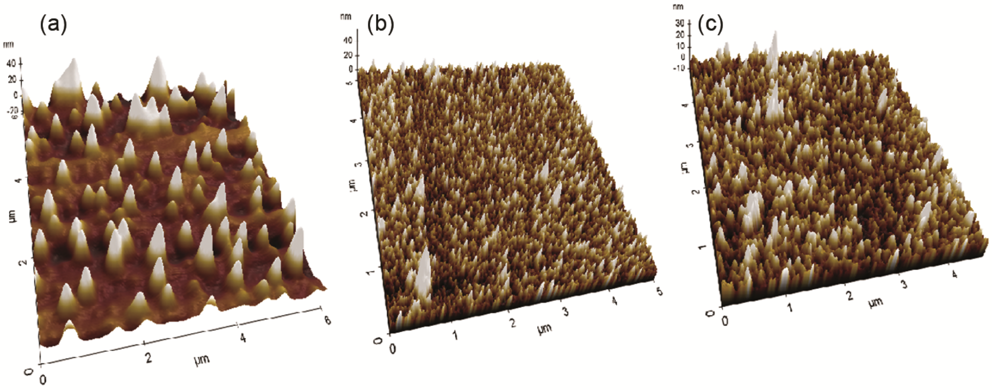


Fig. 2 — AFM micrograph of the Nichrome thin film of various thickness (a) 10 nm (b) 30 nm (c) 70 nm

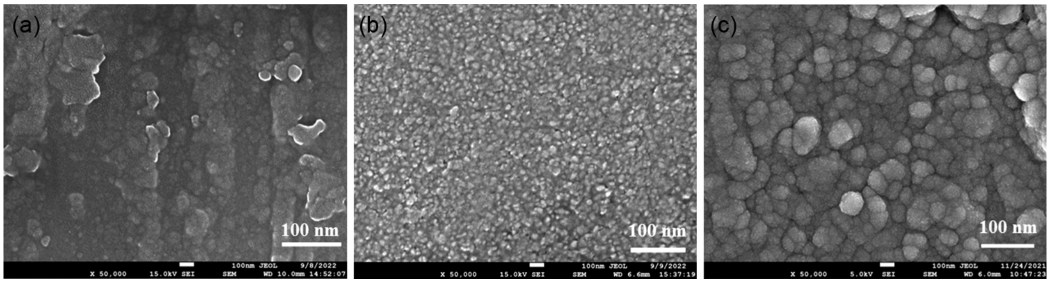
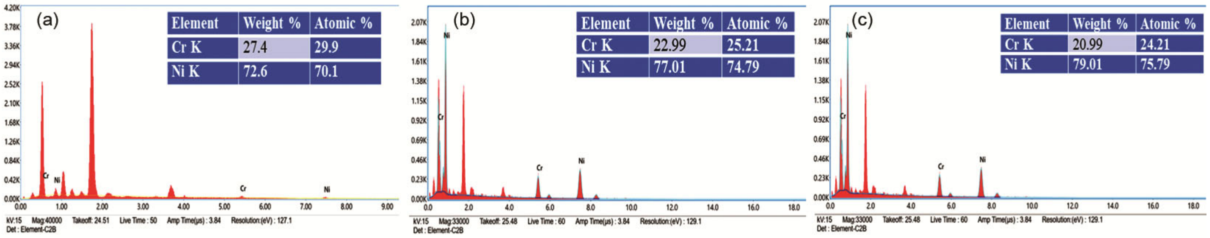


Fig. 3 — SEM images with histogram of Nichrome thin film of various thickness (a) 10 nm (b) 30 nm (c) 70 nm



(a)

Element	Weight %	Atomic %
Cr K	27.4	29.9
Ni K	72.6	70.1

(b)

Element	Weight %	Atomic %
Cr K	22.99	25.21
Ni K	77.01	74.79

(c)

Element	Weight %	Atomic %
Cr K	20.99	24.21
Ni K	79.01	75.79

Fig. 4 — EDAX graph of Nichrome thin film (a) Thickness 10 nm (b) Thickness 30nm (c) Thickness 70 nm

3.3 Surface Morphological Properties

The Morphological analysis of prepared thin film samples was carried out using Scanning Electron Microscopy (SEM). The SEM image of Nichrome thin film of various thicknesses is shown in Fig. 3(a-c). The grain growth is clearly noticed from the SEM images of the Nichrome films. It is observed that the surface morphology is almost smooth and grains are observed.SEM analysis of the films shows microstructures consisting of spherical in case of 30 and 70 nm thin film but there is no clear microstructure shape in 10 nm thickness which is amorphous in nature. Quantitative analysis using Image J software reveals an average grain size of approximately 41.9 nm for the 30 nm film and ~109.3 nm for the 70 nm film. The observed increase in grain size with thickness can be attributed to particle agglomeration tendencies.

Figure 4(a-c) shows the EDX spectrum of Nichrome thin film of different thickness. Two strong peaks were observed corresponding to Ni and Cr indicate a homogenous and smooth coating is achieved. This confirms the successful deposition of Nichrome thin film on the substrate within given composition. This elemental analysis using EDX confirms that films are in accordance with required composition of Nickel and chromium is 80:20. This shows slight variation in 70 nm film thickness but significant difference in composition 72.6:27.4 for 10 nm thin film. Notably, the 10 nm thin film exhibits a significant deviation from the target composition, with uncertainties possibly stemming from measurement instrument precision limitations[44]. In 10 nm thickness Silicon and oxygen peak are present due to glass substrate.

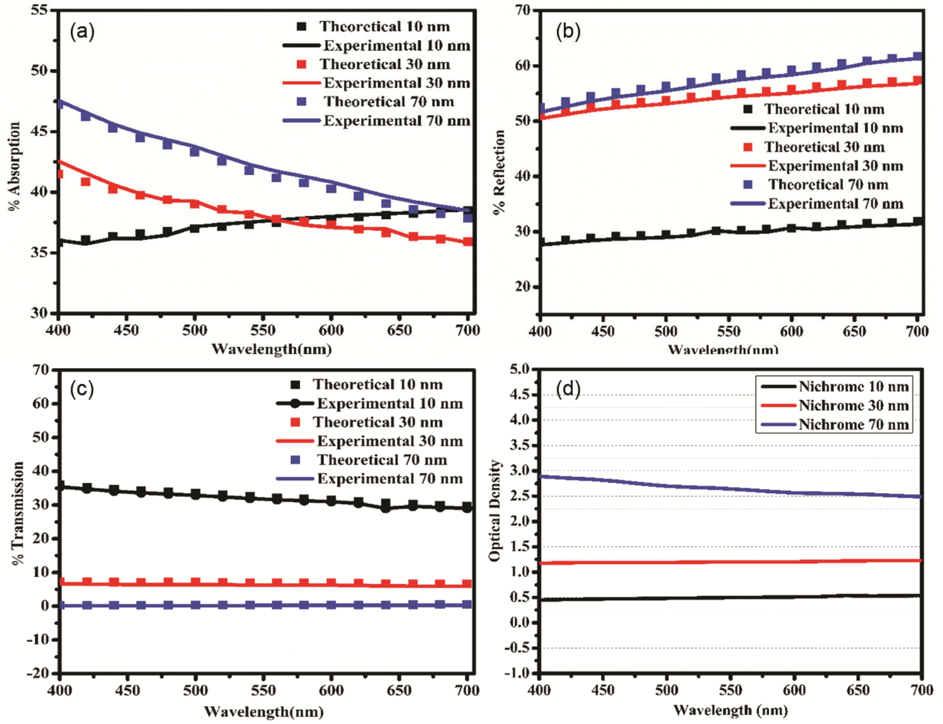


Fig. 5 — Optical measurement of Nichrome thin film of various thickness(a) Absorption spectra (b) Reflection spectra (c) Transmission spectra (d) Optical density

Table 2 — Designed and experimental data of Nichrome thin film at reference wavelength 520 nm

Sample	Transmission (T%)		Reflection(R%)		Absorption(A%)	
	Designed	Experimental	Designed	Experimental	Designed	Experimental
Nichrome (10 nm)	33.02	32.41	29.80	29.28	37.17	37.35
Nichrome (30 nm)	7.08	6.30	54.32	53.64	38.51	38.40
Nichrome (70 nm)	0.367	0.211	57.10	56.21	42.51	43.02

3.4 Optical Properties

The optical properties of Nichrome thin films were investigated using a UV-Vis-NIR Spectrophotometer Varian Cary-5000. Measurements of transmission, absorption, and reflection across the visible region (400-70 0nm) were recorded with a wavelength step of 1nm. Essential Macleod Software was utilized to simulate single-layer Nichrome coatings of thicknesses 10, 30, and 70 nm[45]. These simulations aimed to optimize transmission, reflection, and absorption characteristics by adjusting thickness. Nichrome was modelled as a single layer on a glass substrate, with a ° angle of incidence at a reference wavelength of 520 nm. Experimental results, depicted in Fig. 5, exhibited good agreement with the theoretical model. The comparison between the designed and experimentally deposited films is detailed in Table 2. Notably, these thin films displayed neutral absorption and transmittance within the visible region (400-700 nm). For the 10 nm thickness, an Optical Density (OD) value of 0.5 was obtained. An OD of 0.5 suggests moderate light attenuation, reducing light intensity reaching the sensor by over 68%. This level of attenuation can be advantageous in color sorting applications with bright lighting conditions, enhancing color detection accuracy by preventing overexposure. The variation in Optical Density is ΔOD=0.08.In contrast, the 70 nm thickness film exhibited an OD of 2.7[50]. This indicates that only about 0.2% of the incident light is transmitted through the material, with approximately 99.8% of the light absorbed or blocked. Filters with an OD of 2.7 find utility in applications requiring significant reduction in light intensity, such as laser safety goggles or situations necessitating minimal light for camera or sensor operation to prevent overexposure or glare. The variation in Optical Density is ΔOD=0.44. The range of optical density lies in between 0.1 to 3. The optimal values of optical density for spectrophotometer are in

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Nichrome (70 nm)	0.367	0.211	57.10	56.21	42.51	43.02

Table 3 — Optical parameters of Nichrome thin film at reference wavelength 520 nm

Sample	Optical density(OD)	Abs. Coefficient(α) nm^{-1}	ΔOD (Spectral Variation)	Refractive index(n)		Extinction Coefficient(k)	Transmission variation (in %)
				Present Work	R.R.Willey		
Nichrome 10nm	0.5	0.048	0.08	2.1	2.3	2.02	6.3
Nichrome 30nm	1.2	0.039	0.05	3.1	3.0	1.64	0.77
Nichrome 70nm	2.7	0.038	0.41	3.4	3.5	1.58	0.2

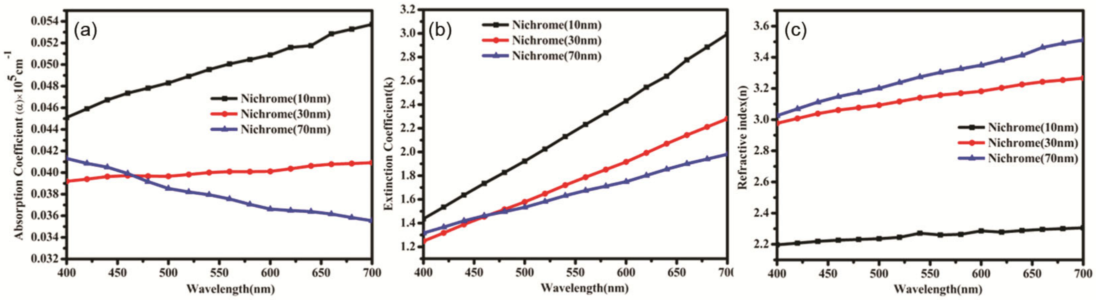

Fig. 6 — Optical measurement of Nichrome thin film of various thickness (a) Absorption coefficient (b) Extinction coefficient (c) Refractive index

range from 0.3 to 0.9[33] and my values are closely related to this. The optical densities of 3.0 or less are ideal for color sorting and separating spectral orders[34]. The obtained optical parameters of Nichrome thin films at the reference wavelength of 520 nm are presented in Table 3.

The absorption coefficient is calculated by using following equation[46]

$$\alpha = \frac{OD}{t} \qquad ...(3)$$

Where OD is Optical density and t is the thickness of thin film.

The Extinction coefficient can be assessed by using the absorption coefficient data[47].

$$k = \frac{\alpha\lambda}{4\pi} \qquad ...(4)$$

The refractive index (n) of nickel thin film is calculated by using this equation[45]

$$n_1 = (n_2)^{1/2}\left(\frac{1 \pm R^{1/2}}{1 \mp R^{1/2}}\right)^{1/2} \qquad ...(5)$$

In the equation where R represents reflectance and n_2 denotes the substrate refractive index, the choice of plus or minus sign hinges on whether the film index surpasses or falls short of that of the substrate. Notably, the absorption coefficient diminishes as thickness increases as shown in Fig. 6. This observation strongly suggests that as the thickness of Nichrome thin films increases, a greater number of photons are absorbed within them[48].

Moreover, the decline in the extinction coefficient with increasing thickness implies a smooth surface for the thin film. Our findings align with SEM data, indicating that our films become denser with increased thickness. This densification likely contributes to the notable elevation in the index of refraction[49] which is closely related to Willey[50] given in Table 3.

4 Conclusion

In the present work we designed Neutral Density Filter in 400 nm-700 nm using thermal evaporation of Nickel and Chromium. We proposed a co-evaporation method to produce thin layer 10, 30 nm and 70 nm thickness with thickness dependence of OD. The computer simulation using Essential Macleod software confirm the designed OD can be produced from 0.5 to 2.7 with just varying the thickness of film which is good for color sorting applications. XRD measurement provide information about crystallite size that is increases with thickness but lattice strain is decreases as thickness increases. Nichrome thin film having cubic structure with space group Pm-3n (223) has successfully deposited without any impurities. AFM confirm the good quality surface with uniform composition confirmed in EDX measurement. FE-SEM image analysis reveals that the grain size is increases with increases of thickness and homogenous coating is achieved.

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Declarations

Conflicts of interest

The authors have no conflicts of interest.

Data Availability

The data that support the findings of this study will be available from the corresponding author upon reasonable request.

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