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Reactively Sputtered $InVO_4$ Thin Films: Effect of Non-Stoichiometric Composition on Photocatalytic Activity

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Abstract: ***Indium-vanadium oxide ($InVO_4$) thin films were deposited on silicon and quartz substrates by reactive direct-current magnetron sputtering. The deposition parameters were optimized to give stoichiometric composition and the films were post-annealed to vary the crystallinity, crystal structure and properties of thin films. Later, by suitably changing the deposition parameters, non-stoichiometry was induced to result in V-excess $InVO_4$ and In-excess $InVO_4$ films after duly annealing. The films were characterized by XRD, UV-Visible spectroscopy and scanning electron microscopy (SEM). At higher annealing temperatures, the V-excess $InVO_4$ contained V_2O_5 nanorods on the film surface while In-excess $InVO_4$ had no special structures on the film surface. Photocatalytic properties of the films under visible-light illumination were evaluated by the reduction of Ag^+ in $AgNO_3$ aqueous solution, the degradation of methylene blue (MB). The relationship among post-annealing temperature, coupled composite nature of the non-stoichimetric compositions and photocatalytic properties was explored in this study.***
Keywords: Indium-vanadium oxide, visible light photocatalyst, reactive sputtering, thin film, non-stoichiometry

I. INTRODUCTION

Photocatalysis – a convenient means of using solar radiation to accomplish and accelerate various chemical reactions is being specially exploited for applications involving energy and environment in the past decade [1-3]. Titanium dioxide (TiO_2) is the single major metal oxide that has been studied in depth for its versatile use in heterogeneous photocatalysis like splitting of water into hydrogen and oxygen, remediation of various organic pollutants that are present in air or water and as pigments in paints and paper industry [4]. One of its surface property, the super hydrophilicity makes it a self-cleaning, anti soiling and anti fogging material when exposed to UV radiation [5]. Apart from TiO_2, many other metal oxides (simple or multi-component) are simultaneously explored [6-8] for their potential photocatalytic activity in the visible range.

The presence of a suitable bad gap is the essential prerequisite for any compound to be a visible light photocatalyst and is satisfied only by some compounds. Multicomponent oxides whose valence band and the conduction band are aptly tuned to suit the photosplitting of water into H_2 and/or O_2, are mainly vanadates, tantalates, niobates, molybdates and wolframites containing one or more of metal atoms In, Bi, Cr, Pb and Ni in them. Kudo et al [9,10] have shown that $In_2O_3(ZnO)_m$ and $BiVO_4$ could produce H_2 or O_2 from methanol/silver nitrate solutions under visible light irradiation. Recently, a single phase Aurivillius type perovskite, $PbBi_2Nb_2O_9$ has been found [11] to be exhibiting high quantum yield of 29 % in oxygen production from water containing $AgNO_3$. Jinhua Ye et al have correlated the crystal and electronic structures of a new series of photocatalysts $InMO_4$ ($M=V^{5+}$, Nb^{5+}, Ta^{5+}) [12]. A member of this series, $InTaO_4$ when doped with Ni ($In_{1-x}Ni_xTaO_4$, x = 0-0.2) could split water into stoichimetric amounts of oxygen and hydrogen under visible light irradiation with high quantum yield [7]. The vanadate in the series, $InVO_4$, was found to posses a much narrower band gap of about 1.9 eV compared to the other two [12]. The band gap in $InVO_4$ is narrowed due to the 3d orbital of vanadium in the conduction band as compared to 4d and 5d of Nb and Ta respectively. Pure $InVO_4$ and NiO_x – loaded $InVO_4$ powders prepared by solid-state method could efficiently split water to evolve H_2 and O_2 under visible light illumination ($\lambda >$ 420 nm) [13]. Nanoparticles of $InVO_4$ prepared by using a heteronuclear complex as precursor [14], showed higher photocatalytic activity compared to the sample obtained by conventional solid state reaction. The performance also depended on the shape of $InVO_4$ nanostructure [15] and the porosity of material when it is in the form of a thin film [16].

The problems associated with the removal of suspended fine particles during or after photocatalysis prompt us to use thin films instead of powdered samples. However, not much work is reported on the photocatalysis of $InVO_4$ thin films, specially obtained by DC magnetron sputtering. Thin films of $InVO_4$ and mixed In/V oxides obtained by sol-gel and RF magnetron sputtering respectively are studied for their potential use in electrochromic devices and as insertion electrodes for Li batteries [17, 18]. Theoretical calculations suggested that the band gap of $InVO_4$ is markedly influenced by the V environment around O atom [19]. In this

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context, it is interesting to investigate thin films of $InVO_4$ for the possible enhancement of photocatalytic activity when one of the components is in excess. In the present paper, we propose to investigate the photocatalytic properties of pure stoichiometric $InVO_4$ films, obtained by reactive sputtering and also the effect of non-stoichiometry on the photocatalytic activity.

II. EXPERIMENTAL DETAILS

A. Preparation of $InVO_4$ films

Indium-vanadium oxide ($InVO_4$) thin films were deposited by reactive unbalanced magnetron DC sputtering. The sputtering system allows independent control of several critical deposition parameters and the details of the system were described elsewhere [20]. The targets used are vanadium metal and In_2O_3 powder, which are maintained at different power values of DC (for V metal) and RF (for In_2O_3 powder) to get four combinations of parameters (P1, P2, P3 and P4). The ratios of power values for V: In_2O_3 are 200 W : 200W (P1); 200 W : 80 W (P2); 300 W : 150 W (P3); and 300 W : 100 W (P4) respectively. The distance between the substrate holder and the targets was 50 mm. The chamber was evacuated by a mechanical pump (ALCATEL-2033SD) and a turbo-pump (ALCATEL-ATP400HPC). The flow rates of oxygen and argon were maintained at 13 and 37 sccm respectively. The base pressure is 8.6 x 10^{-4} Pa (6.5 x 10^{-6} Torr) and the working pressure is increased to 8 x 10^{-1} Pa (6 x 10^{-3} Torr). The substrates used to deposit the films are polished N-type Si wafers (1 1 0) of thickness 450-575 µm. The substrate temperature was 150 ^{0}C and was rotated at 12 rpm. The as deposited films were amorphous and hence annealed at temperatures 400, 500, 600 and 700 ^{0}C respectively for 2 hrs each.

Based on the compositional analysis of thin films obtained with different target powers as deposition parameters, attempts were made to induce non-stoichimetry in the composition to obtain minor In-rich and V-rich $InVO_4$ samples. For non-stoichiometric compositions a set of different target powers P5 (V=200W, In=90W; V-rich $InVO_4$) and P6 (V=245W, In=150; In-rich $InVO_4$) are followed. The values of target powers are based on a preliminary experiment involving a calibration curve of V-target power for a fixed target power of In_2O_3 target and the corresponding V at % in the film.

B. Characterization

The crystallinity of the films was investigated by a Bruker D8 Advance X-ray diffractometer (XRD) operating with Cu Kα radiation at 40 kV and 100mA. Raman spectra for the films are recorded on RENISHAW 1000B spectrometer. The film thickness and roughness were measured by a surface profilometer (Stylus Surface profiler, Veeco (USA), Dektak series 3). The absorption spectra were obtained by using Hitachi 3300H UV-Visible spectrophotometer. The surface morphology and the composition of the films are characterized by Scanning Electron Microscopy (SEM) and Energy Dispersive Spectroscopy (EDS) using Hitachi 3500H SEM.

C. Photocatalytic activity studies

The photocatalytic activity was measured (i) by the reduction of Ag^+ ions in 0.1M $AgNO_3$ to Ag metal, upon irradiation of UV & visible light independently, (ii) by monitoring the degradation of 5 ppm MB aqueous solution (Riedel-de Haen) under irradiation of visible light source with time. The UV illumination was carried out using an 8W fluorescent light (UV Itech-LF104L) with the wavelength distribution being around 330-380 nm and the maximum intensity at 350 nm. The distance between the UV light and the sample was 10 cm producing an intensity of 3.2 mW/cm^2 measured by the power meter (MELLES GRIOT Broad band Power/Energy meter 13PEM001). The visible light illumination was carried out using a fluorescent lamp (Hitachi-FML27EX-N) with a wavelength distribution around 400-700 nm and the maximum intensity at 550 nm, producing a power density of 1.5 mW/cm^2 in a distance of 8 cm between the visible light and the sample. The reduction of Ag^+ from $AgNO_3$ solution was studied by taking the SEM pictures before and after carrying out the experiment and estimating the metallic silver formed.

III.RESULTS AND DISCUSSION

A. Crystal structure and crystallinity of films deposited with P1, P2, P3 and P4

The as deposited films (obtained for the parameters P1, P2, P3 and P4) were found to be amorphous and hence they were annealed at temperatures 400, 500, 600 and 700 ^{0}C for 2 hrs each (post-annealing). Structural studies [21] have shown that $InVO_4$ belongs to the class of orthovanadates (RVO_4) and can crystallize in both orthorhombic Cmcm phase ($InVO_4$–III) when annealed at high temperature and in a meta stable monoclinic low-temperature C2/m phase ($InVO_4$-I). The XRD patterns of post-annealed films deposited with P2 show that the films are crystallized in orthorhombic phase of $InVO_4$ at temperatures 500 - 700 ^{0}C (Figure 1). Though the films showed a small amount of VO_2 impurity at post-annealing temperatures of 500, 600 ^{0}C, a pure stochiometric film was obtained as annealing temperature is increased to 700 ^{0}C. The compositional analysis obtained from EDS (Table 1) also showed

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that the empirical formula of the thin films deposited with P2 at 700 ^{0}C is very close to $InVO_4$. However, the films deposited with P1, P3 and P4 contained a mixture of monoclinic and orthorhombic phases of $InVO_4$ with VO_2 and/or In_2O_3 as considerable phase at all the post-annealing temperatures (the XRD patterns are not shown here).

For films deposited with P1 the composition analysis obtained from EDS (Table 1) showed an In/V atomic ratio far from unity and neither the monoclinic nor the orthorhombic phase of $InVO_4$ is formed as major phase. The compositional analysis of post annealed films deposited with P3 and P4, show an empirical relation better than the films of P1 in terms of their atomic ratios (Table 1) but had mixed phases. Artuso et al [22] have taken three targets of In_2O_3/V_2O_5 (mixed and cold pressed) with molar ratios of 1.0, 0.5, 0.25 and sputtered at RF power of 200 W. Their XPS results showed that the In/V and O/V ratios in the films are close to the target In/V molar ratios of 1.0 and 0.5. Since these films are not heat treated after sputtering, the films contained an admixture of both amorphous and crystalline $InVO_4$ with vanadium deficiency at the surface. Sol-gel processed $InVO_4$ thin films [23] also ended up with a mixture of monoclinic $InVO_4$-I phase and orthorhombic $InVO_4$-III phase after they

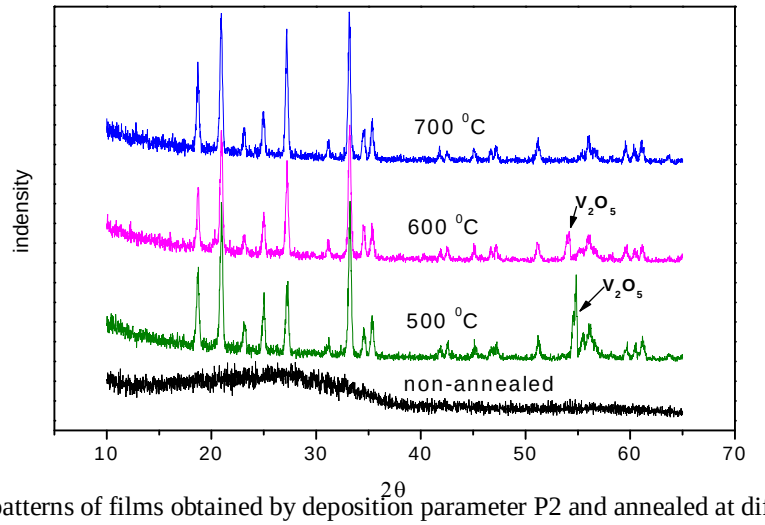

Fig. 1 Powder XRD patterns of films obtained by deposition parameter P2 and annealed at different temperatures

TABLE I
FILM DEPOSITION PARAMETERS AND COMPOSITION ANALYSIS

Parameter	Power ratio of V metal (DC) : In_2O_3 (RF)		Atomic ratio from EDS In/V	O/V
P1	200 W	200 W	2.73	17.23
P2	200 W	80 W	0.99	6.25
P3	300 W	150 W	1.08	3.89
P4	300 W	100 W	0.82	6.89

were thermally treated at 500 ^{0}C for 1 hr. Therefore the deposition parameters and the right annealing temperature are very crucial for obtaining a stoichiometric thin film of single phase $InVO_4$. Eventually, in the present experiment, the deposition parameters as P2 (V=200W, In=80W) are optimal for obtaining a stoichiometric $InVO_4$ film at post annealing temperature of 700 ^{0}C.

B. Crystal structure and crystallinity of films deposited with P5 and P6

The powder XRD patterns of thin films deposited with parameters P5 and P6 and annealed at different temperatures are studied in detail. The films remained amorphous until the annealing temperatures were 400 ^{0}C (P5) and 500 ^{0}C (P6) respectively. The crystallinity improved upon annealing further and at 600 ^{0}C an orthorhombic $InVO_4$ phase with an excess of V_2O_5 was formed with deposition parameters of P5. However, with deposition parameters P6 a monoclinic $InVO_4$ with an impurity of In_2VO_5 is formed when annealed at 600 ^{0}C.

C. Optical absorption

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The UV-Vis absorption spectra of post-annealed films deposited by parameters P5 (V-rich $InVO_4$) and P6 (In-rich $InVO_4$) are recorded and for P5 it is shown in figure 2. As the annealing temperature is increased the absorption edge of the films shifted from lower wave length to higher wave lengths of visible region for the V-rich films. But for the In-rich films the trend was opposite i.e. a blue shift was observed with annealing temperature. It is well known [24] that excess of indium increases the transparency of the films and an excess of V_2O_5 increases the absorption near 500 nm (red region of visible light) and hence the observation of red shift (V-rich) and blue shift (In-rich) in the present non-stoichiometric films. The absorption edge changed from ~ 480 nm to ~ 650 nm in the V-rich $InVO_4$. Similarly the blue shift observed in In-rich $InVO_4$ is from ~ 480nm to ~ 380 nm. The shift of absorption edge to higher wavelengths is also ascribed to higher crystallinity of $InVO_4$-III phase (orthorhombic) with increased annealing temperature. The profile of absorption for V-rich $InVO_4$ thin film is similar to the earlier report on $InVO_4$ powder [13] and extending further into the visible region due to excess V_2O_5.

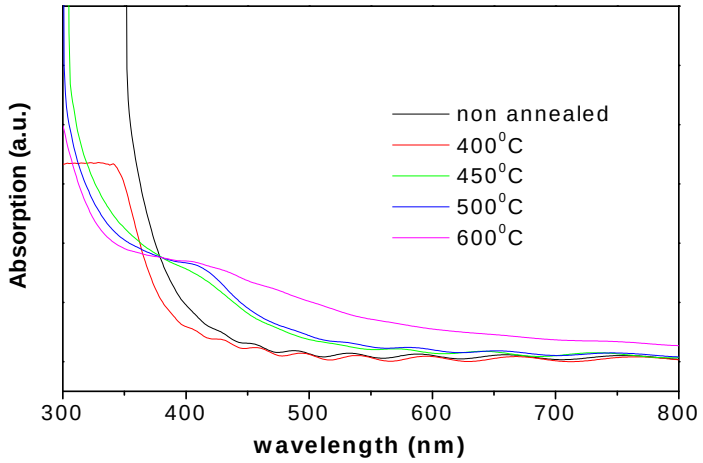

Fig.2 UV-Vis absorption spectra of post-annealed films deposited by parameters P5 (V-rich $InVO_4$)

D. Surface morphology of non-stoichiometric $InVO_4$ films

The SEM images of V-rich and In-rich $InVO_4$ at different annealing temperatures are shown in figure 3. It is interesting to know that the surface of V-rich $InVO_4$ is covered with nanorod structures at 500 ^{0}C (Fig. 3a) annealed film and it is further covered with a thick layer of embedded nanorods in the film annealed at 600 ^{0}C. To identify the composition of nanostructures formed on surface, a part of the film is analyzed by TEM. It was found from the diffraction pattern that the nanostructures are of V_2O_5 phase with orthorhombic structure. This result further confirms the conclusions deduced from XRD. However, the reasons for the growth of excess V_2O_5 into nanorods on the surface are unclear now. In contrary, the surface of In-rich $InVO_4$ at all annealing temperatures is smooth except for a flake like appearance in the film annealed at 600 ^{0}C.

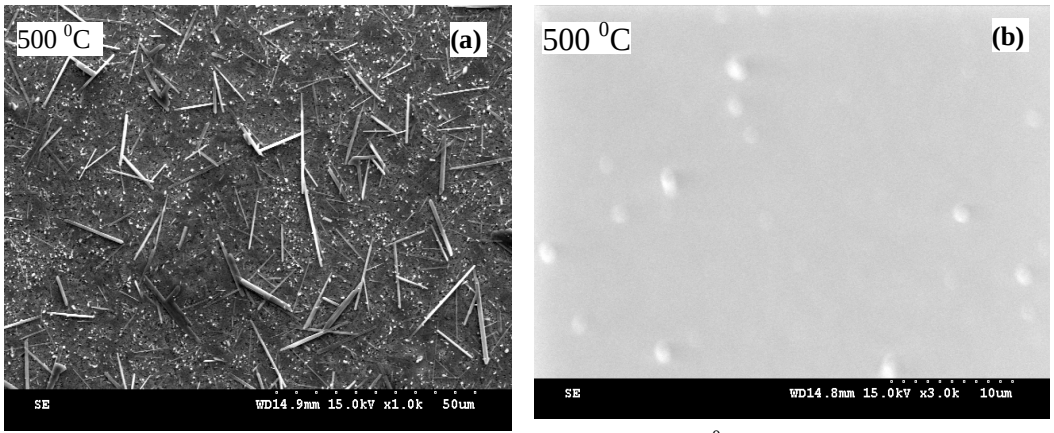

Fig. 3 SEM images of V-rich (a) and In-rich $InVO_4$ (b) at 500^{0}C annealing temperature

E. Photocatalytic activity

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1) *Silver ion reduction:* Both V-rich and In-rich $InVO_4$ thin films annealed at different temperatures are dipped in $AgNO_3$ solution and illuminated with visible light. The surface morphology of the films, after the reduction of silver ions (Ag^+) is shown in the SEM images presented in figure 4. The quantities of reduced silver (metallic Ag) obtained from EDS are given in table 2, for the films post-annealed at all temperatures. The SEM pictures after reduction show some bright spots in V-rich $InVO_4$ which correspond to reduced silver (Fig. 4a). No such observation was made in In-rich $InVO_4$ films (Fig. 4b). Moreover, the extent of bright spots or the amount of reduced silver in V-rich $InVO_4$ increases with annealing temperature. It can also be observed that the reduced silver has aligned along the nanorods of V_2O_5 that were formed on V-rich $InVO_4$ films.

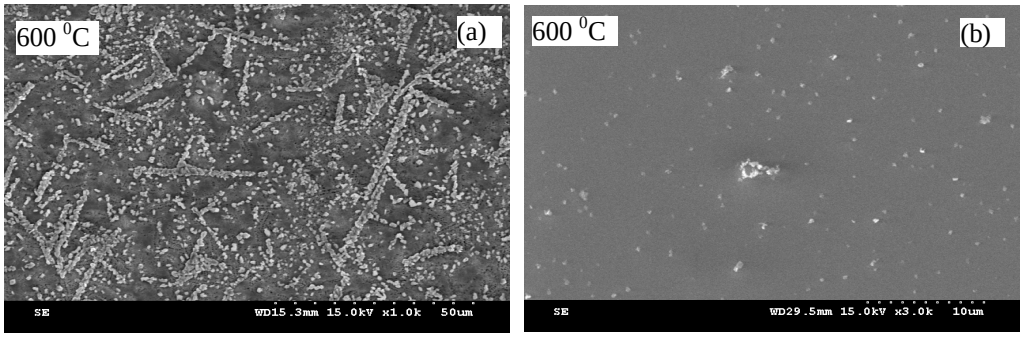

Fig. 4 SEM images of silver ion reduction under visible light for V-rich (a) and In-rich $InVO_4$ (b) films

TABLE III
QUANTITIES OF SILVER ION REDUCED BY POST ANNEALED FILMS OF P5 & P6

Post annealing temperature	Silver ratio for P5 (V-rich $InVO_4$)		Silver ratio for P6 (In-rich $InVO_4$)
	Visible light	UV light	Visible light/ UV light
400 °C	0.019	0.026	<< 0.019
450 °C	0.020	0.024	<< 0.019
500 °C	0.044	0.054	<< 0.019
600 °C	0.184	0.233	<< 0.019

The increased photocatalytic reduction of silver in V-rich $InVO_4$ is perhaps due to the coupled nature of V_2O_5 and $InVO_4$ in the film that behaves as a composite catalyst. Usually V_2O_5 is strongly oxidizing in water. But when it is in contact with a visible light photocatalyst $InVO_4$, the photo generated electrons are inclined to migrate towards V_2O_5 nano rods and reduce Ag^+ at the surface of V_2O_5 nanorods. So the formed silver particles mostly appear to be aligned on the edge of V_2O_5 nanorods. The increased amount of silver on the films annealed at higher temperatures indicates that the crystallinity of both $InVO_4$ and the V_2O_5 contribute to the photocatalytic performance of coupled oxides.

Similar results were obtained when the reduction was carried out under UV light irradiation. However, the percentage reduction of Ag^+ increased with the annealing temperature of thin films. This was evident from the amount of silver formed after reduction on each film annealed at different temperature. Photocatalytic activity of $InVO_4$ powder was also found to be better [13] under UV irradiation than visible light, in terms of the H_2 evolved during water splitting. The photocatalytic activity depends on the wavelength (λ) of the light and generally the activity decreases with increased wavelength [25].

The failure of In-rich $InVO_4$ to reduce Ag^+ ions under visible light illumination demonstrates the catalytic inactivity of mixed monoclinic $InVO_4$ and In_2VO_5 phases of the film. This indicates a drastic effect of non-stoichiometry towards photocatalysis.

2) *Degradation of methylene blue (MB):* The photocatalytic activity of V-rich $InVO_4$ was also evaluated by measuring the decomposition rates of methylene blue (MB). Figure 5 shows the visible light induced photo degradation of methylene blue on the post-annealed films. The change in the concentration of MB was studied for 25 hrs. The figure shows a general decrease in the concentration of MB with time for all films. However, the slope of the plot which generally indicates the extent of reduction is not linear. Perhaps the kinetics of degradation of MB might not follow simple first order reaction. The photocatalytic activity was not linearly dependent on the post annealing temperature either. After 25 hrs, the film annealed at 450 °C showed highest degradation of MB.

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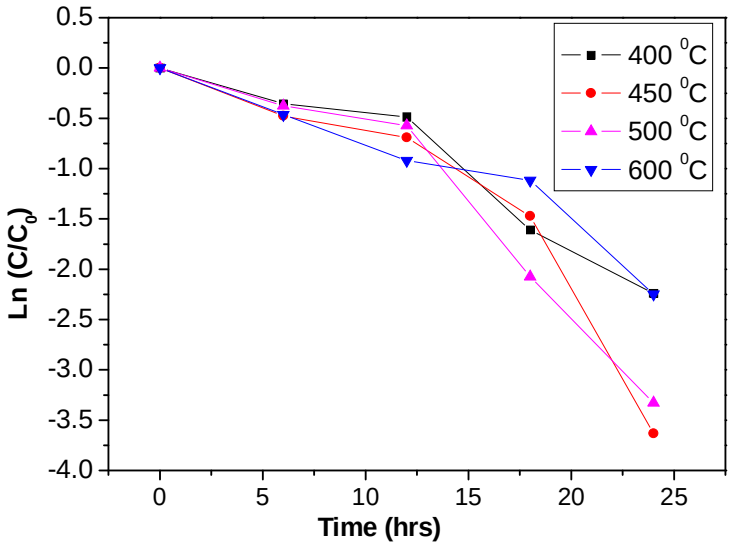

Fig. 5 Visible light induced photo-degradation of Methylene blue on post annealed films of V-rich $InVO_4$

IV. CONCLUSIONS

The stoichiometry of reactively sputtered thin films of Indium-vanadium oxide ($InVO_4$) depended on the deposition parameters. The crystallinity of films could be improved by post-annealing. By changing the power values of targets, V metal and In_2O_3, a slightly non-stoichiometric compositions with V-rich and In-rich $InVO_4$ films were deposited. The V-rich $InVO_4$ film contained nanorods of V_2O_5 on the film surface. In-rich $InVO_4$ has In_2VO_5 as additional phase. The absorption edge of V-rich $InvO_4$ showed red shift with annealing temperature while In-rich $InVO_4$ blue shift. The photocatalytic properties of V-rich $InVO_4$ films are far superior to the In-rich $InVO_4$ films and the activity increased with post-annealing temperature. The coupling of V_2O_5 with $InVO_4$ for efficient charge separation upon visible light illumination is probably the main factor for enhanced photocatalytic performance of V-rich $InVO_4$. Non-stoichiometric In-rich $InVO_4$ is photo catalytically inactive compared to V-rich $InVO_4$.

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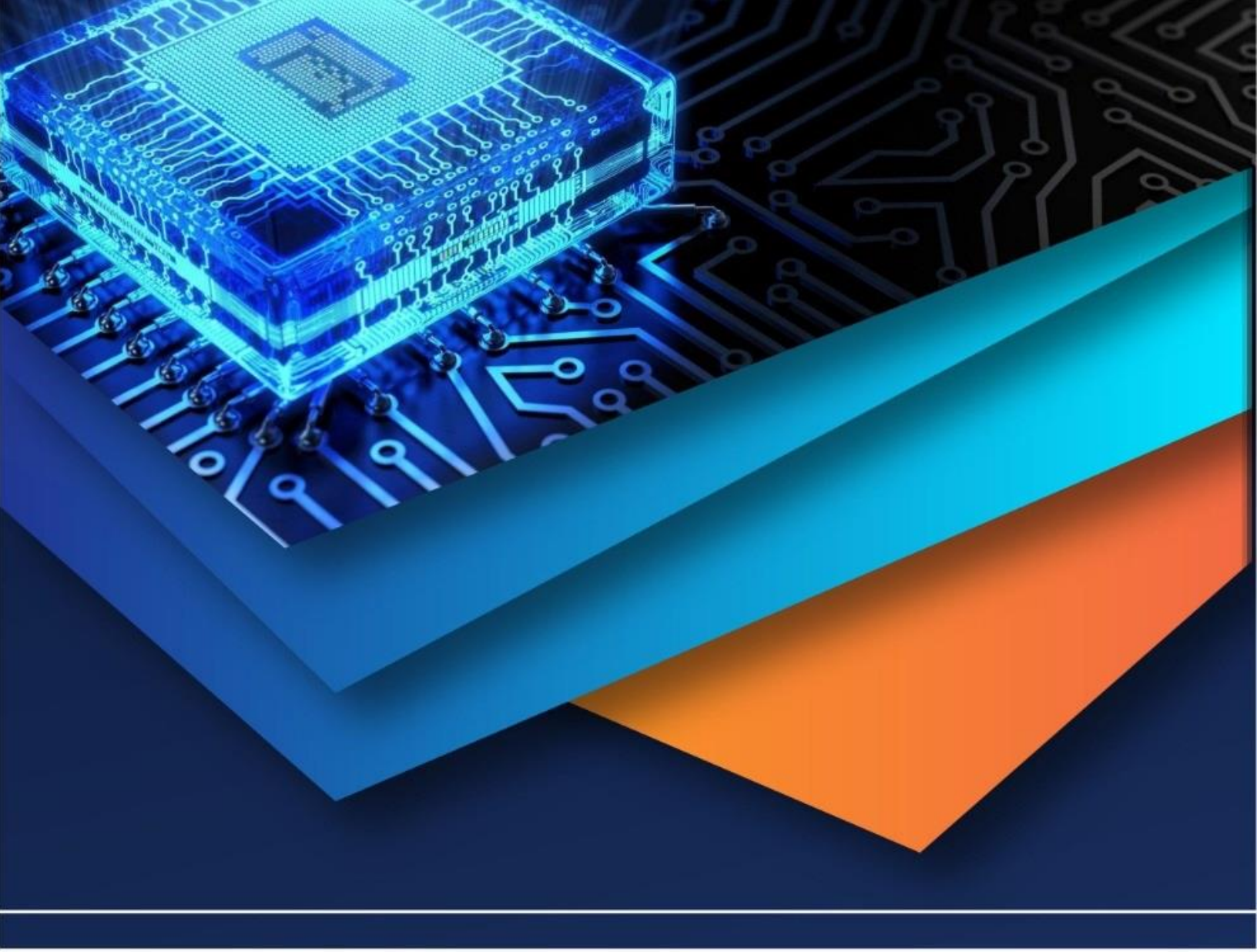

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