

INTERNATIONAL RESEARCH FELLOWS ASSOCIATION'S
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February-2019 Special Issue - 110 (I)

Physics

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Surface Activated Cr₂O₃ Based Thick Film for Ammonia Gas Sensing

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Abstract:

Pure chromium oxide (Cr₂O₃) nanoparticles powder was prepared by chemical co-precipitation method. The structural behaviour of Cr₂O₃ nanoparticles was examined by X-ray diffraction. Thick films of pure chromium oxide were prepared by the screen printing technique. The Fe₂O₃-modified films obtained by dipping pure Cr₂O₃ thick films into an aqueous solution of ferric chloride (FeCl₃) for 3min. and fired at 500^oC for 30 min. Pure and Fe₂O₃ modified Cr₂O₃ thick films were examined by FE-SEM and EDX. Thick film of pure chromium oxide was almost insensitive to ammonia gas. Thick film of Fe₂O₃-modified Cr₂O₃ (3 min dipped) was observed to be highly sensitive to ammonia at 100^oC. The instant response and fast recovery are the main features of this sensor. The effects of surface modification on the gas response, selectivity, response time and recovery time of Cr₂O₃ based thick film gas sensor in the presence of NH₃, Cl₂, LPG, CO₂, H₂S and C₂H₅OH gases were studied and discussed.

Keywords: Chromium oxide, X-ray diffraction, Ammonia gas, Gas response.

Introduction

The metal oxides based semiconductor gas sensors are playing an important role in the detection of toxic pollutants in air and to control the industrial processes. It is observed that when the sensors are exposed to the atmosphere, the chemical species to be detected in the atmosphere can enter in to the interface of the p-n junction. So there are changes in electrical properties at the junction [1-3]. Therefore, the gas sensors were synthesised by using, ZnO, SnO₂, TiO₂, WO₃, Cr₂O₃, etc. materials in pure and modified forms to detect toxic, harmful, flammable and explosive gases [4,5] in the environment. Cr₂O₃ has a Hexagonal-Rhombic corundum crystal structure showing p-type semi- conductivity and has a high melting temperature (~ 2300^oC). In the recent years, p-type Cr₂O₃ is useful for sensor applications as it has an energy band gap of ~3.4 eV and widely been used in variety of applications, such as, catalytic reactions, optical coating and infrared sensors [6-9]. It has been studied that Cr₂O₃ [10-13] is used as a gas-sensing materials. Cr₂O₃ is the transition-metal oxide and the transition-metal oxides are more sensitive to the change of outside ambient and these types of oxides could be more preferable for the use in gas sensors [14]. It has been found that a pure Cr₂O₃ thick film has poor gas sensitivity to reducing gases but Fe₂O₃ modified Cr₂O₃ thick film is the most sensitive to LPG, C₂H₅OH, NH₃ and Cl₂ gases [12].



The particle size plays an important role in gas sensing. The reduction in grain-size is one of the prominent factors for enhancing the gas sensing properties of semiconducting oxides. Many preparation techniques for synthesis of Cr₂O₃ nanoparticles are described, such as hydrothermal reduction [15], solid thermal decomposition [16], urea method [17], and mechanochemical reaction [18], etc. In present work, co-precipitation methods (chemical route methods) were adopted, as they are easy, suitable and economically low cost to synthesize Cr₂O₃ nanostructures.

The aim of the present work is to fabricate the ammonia sensor by modifying pure Cr₂O₃ thick films, which is able to detect ammonia at trace levels. Among the various metal oxide additives tested, Fe₂O₃ is outstanding in promoting the sensing properties of the Cr₂O₃ sensor for ammonia. The present paper reports the structural, morphological and gas sensing properties of pure and Fe₂O₃ modified Cr₂O₃ based thick films.

Experimental Details

Preparation of Nanocrystalline Cr₂O₃ Powders

All chemicals were of analytical grade and used directly without further purification. Nanocrystalline Cr₂O₃ powders were synthesized by chemical precipitation method. The details regarding preparation of nanocrystalline Cr₂O₃ was already published in our earlier publication [19]. The synthesized Cr₂O₃ nanostructure product was used for further study.

Fabrication of thick films and Fe₂O₃ modified Cr₂O₃ thick films

Thick films of pure chromium oxide were prepared by the screen printing technique. In this process thixotropic paste was formulated by proper method and then the thixotropic pastes were screen printed on a glass substrate in desired patterns. The films prepared were fired at 500^oC for 12 h. Prepared thick films termed as pure Cr₂O₃ thick films. The surfaces of pure Cr₂O₃ thick films were modified by dipping them into 0.01M aqueous solution of FeCl₃ for 3 min. The FeCl₃ dispersed on the film surface was oxidised to Fe₂O₃ in firing process and sensor elements with Fe₂O₃ on the surface of Cr₂O₃ thick films were obtained. These surface modified thick films are termed as Fe₂O₃ modified Cr₂O₃ thick films.

Results and discussion

X-ray diffraction

Fig. 1 shows X-ray diffraction (XRD) pattern of synthesized pure Cr₂O₃ powder sample. The observed peaks are matching well with JCPDS reported data of pure Cr₂O₃ with hexagonal in structure (JCPDS card no.70-3766). The characteristic peaks observed in the spectrum are higher in intensity which indicates that the as-synthesized samples are of good crystalline in nature. The domain size of the crystal can be estimated from the full width at half maximum (FWHM) of the peaks by means of the Scherrer formula [20]. The average crystallite size (D) was estimated from the Debye-Scherrer's equation: $D = 0.9 \lambda / \beta \cos \theta$; where λ is the wavelength of X-rays (1.54056 Å), β is the FWHM of the peak in radians, θ is the diffraction angle at which the full width at half maximum (FWHM) measured and $k = 0.9$, is Scherrer constant. The average crystallite size of the synthesized Cr₂O₃ nanoparticles calculated from (110) peak was approximately 26 nm.

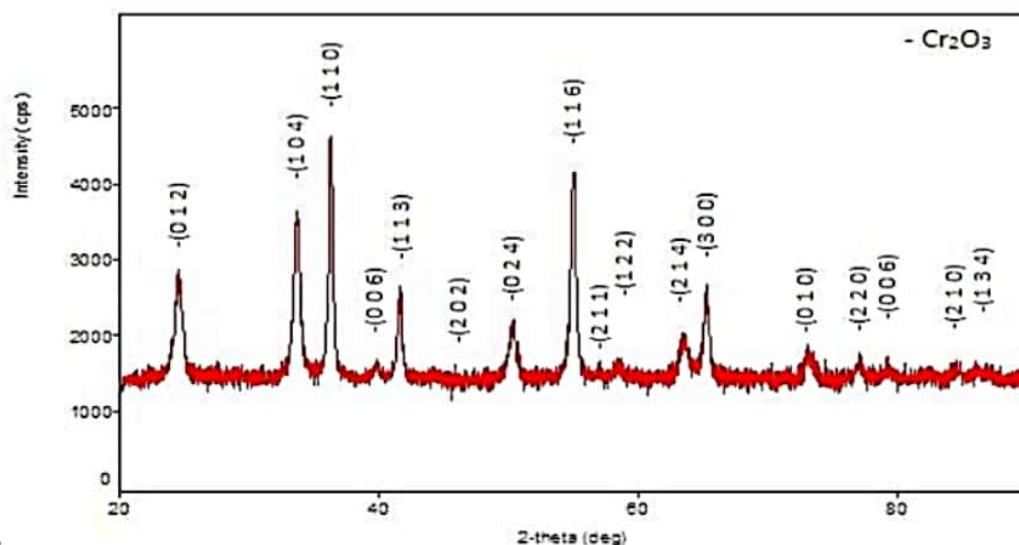


Fig
Scanning electron microscopy

Fig. 2 (a-b) depicts the FE-SEM images of the pure Cr₂O₃ film and Fe₂O₃ modified Cr₂O₃ thick film (3 min dipped). Fig. 2 (a) shows the FE-SEM image of the pure Cr₂O₃ film, which consists of randomly distributed grains with larger size and shape distribution. The average size of Cr₂O₃ grains are approximately 37 nm. The appearance of the film looks porous, which supports the adsorption and desorption type of gas sensing mechanism. The nano scaled grains exhibit high surface to volume ratio.

Fig. 2 (b) depicts the microstructure of Fe₂O₃ modified Cr₂O₃ thick film for 3 min dipped consists of large number of almost similar sized grains of Fe₂O₃ associated with the comparable sized grains of Cr₂O₃. The film consists of grains with sizes ranging from 19 nm to 33 nm distributed non-uniformly. The appearance of the film looks porous in nature (not looks masked). Thus increase in surface to volume ratio, which enhances the large number of gas adsorption upon exposure giving larger gas response. This film is observed to be the most sensitive to 100 ppm NH₃ gas at 100 °C due to its porous nature.

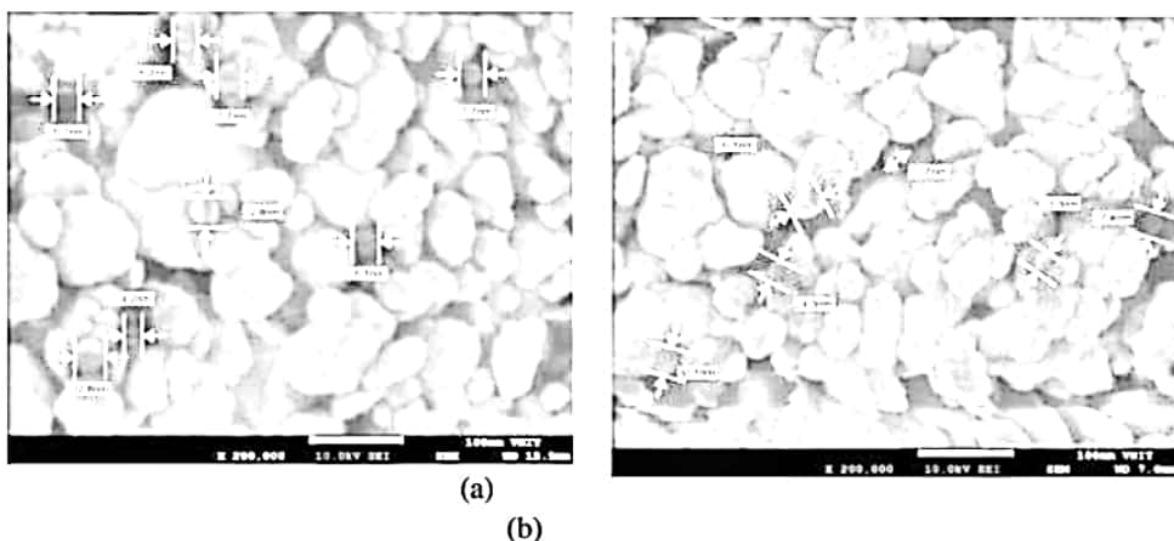


Fig. 2: FE-SEM microstructures for (a) Pure Cr₂O₃ thick film (b) Fe₂O₃ modified Cr₂O₃ thick film (3 min dipped)

Quantitative Elemental Analysis (EDX)

The quantitative elemental composition of the pure and Fe₂O₃ modified Cr₂O₃ thick films were analyzed using an Energy Dispersive Spectrometer (EDS). Fig.3 (a-b) represents the EDS patterns of pure and Fe₂O₃ modified Cr₂O₃ thick films (3min dipped). In chromium oxide, stoichiometric mass percentage of chromium and oxygen are 68.420 and 31.580 respectively.

The prepared powder of pure Cr₂O₃ is excess in oxygen, which increases its p-typeness characteristic. It leads to semiconducting nature of the synthesized pure Cr₂O₃. Excess or deficiency of the constituent element leads to the semiconducting nature of the material. Also, the mass % of Cr and O in each activated samples are not as per the stoichiometric proportion and all samples are observed to be oxygen deficient or excess in chromium. Thus, the maximum numbers of electrons are free to conduct the current and electrons behave as the majority current carriers. This enhances n-typeness of activated Cr₂O₃. Thus, dipping process is the simple and low cost technique to activate the surface of the film. This forms heterojunctions on the surface of the film which increases the resistivity.

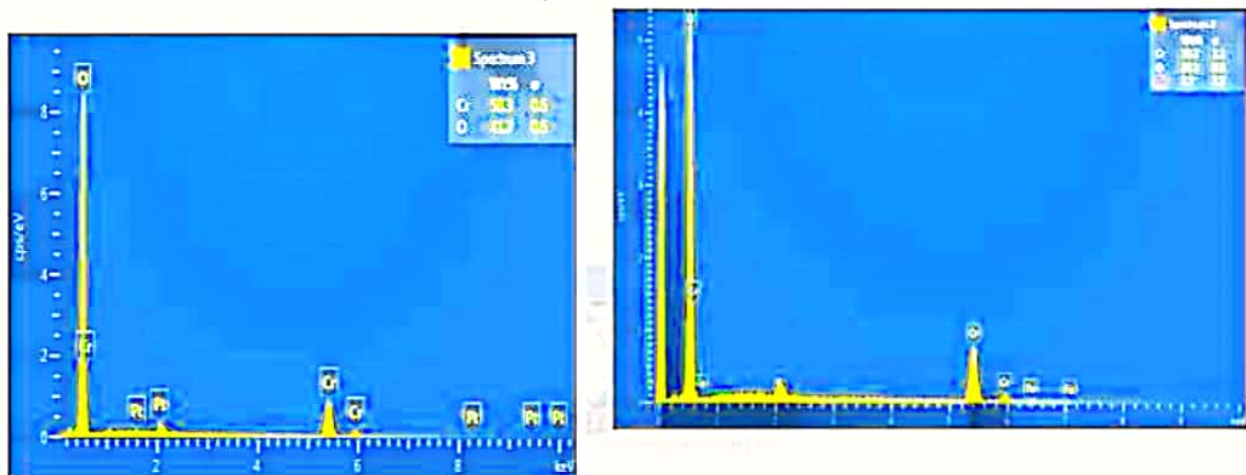


Fig.3: EDS patterns for a) pure Cr₂O₃ thick film b) Fe₂O₃ modified Cr₂O₃ thick film (3 min dipped)

I-V characteristics pure and Fe₂O₃ modified Cr₂O₃ thick films

Fig.4 depicts the I-V characteristics of pure and Fe₂O₃ modified Cr₂O₃ thick films. It is clear from the symmetrical I-V characteristics that the silver contacts on the film were ohmic in nature. Fig. 4 also shows that the conductivity of pure Cr₂O₃ film is larger than Fe₂O₃ modified Cr₂O₃ thick films.

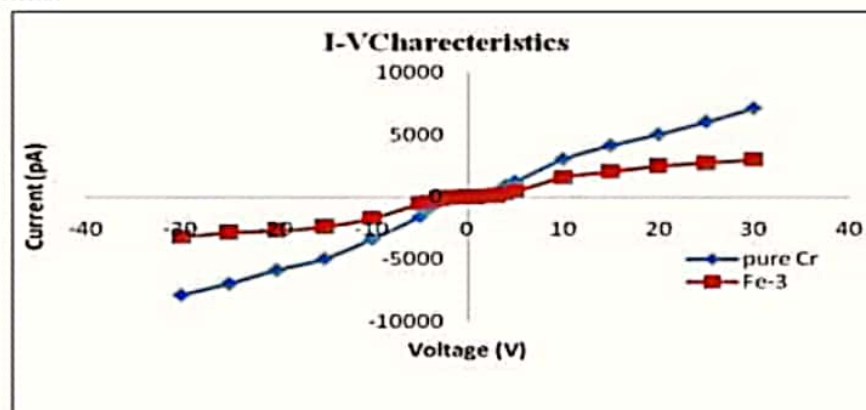


Fig.4: I-V characteristics of pure and Fe₂O₃ modified Cr₂O₃ thick films at room temperature.
Gas Sensing Performance of Pure and Fe₂O₃ modified Cr₂O₃ thick films

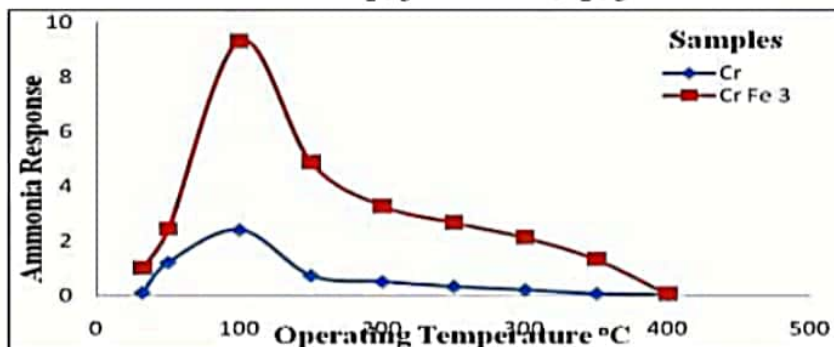


Fig 5: Variation of gas response of pure and Fe₂O₃ modified Cr₂O₃ thick films with operating temperature

Fig. 5 depicts the gas response of pure and Fe₂O₃ modified Cr₂O₃ thick films versus operating temperature. The gas response of pure and Fe₂O₃ modified Cr₂O₃ thick films to 100 ppm NH₃ were investigated at various operating temperatures ranging from room temperature to 400°C. Fig. 5 also shows that the gas response to NH₃ gas goes on increasing with operating temperature, reaches to a maximum at 100°C and then decreases with the further increase of operating temperature. As we know that, response to a NH₃ gas is generally depends on the number of oxygen ions adsorbed on the surface of the film with a target gas. If the film surface chemistry was favourable for adsorption, response and selectivity would be enhanced. In the case of pure Cr₂O₃ thick film, oxygen adsorption seems to be poor and hence it shows poor response to NH₃. So, to improve and enhance the sensing performance of pure Cr₂O₃, it is essential to modify pure Cr₂O₃. It is also clear from figure that Fe₂O₃ modified Cr₂O₃ thick film at 3 min dipping time gives highest response to 100 ppm NH₃ at 100°C. When the optimum amount of Fe₂O₃ (3 min dipping) is dispersed on the surface of Cr₂O₃ thick film, the Fe₂O₃ grains would be distributed uniformly throughout the surface film. These Fe₂O₃ grains form potential barrier (p-n heterojunctions) with Cr₂O₃. Due this potential barrier, sensor element offers high resistance. So such amount of high resistance would be sufficient to promote the catalytic reaction effectively. These overall changes lead to high gas response when this type of sensor element expose to NH₃ gas.

Fig. 6 depicts the selectivity of all, pure and Fe₂O₃ modified Cr₂O₃ thick films towards LPG, C₂H₅OH, CO₂, NH₃, H₂S and Cl₂ for 100 ppm concentration at 100°C. It is observed from figure that the 3 min. dipped Fe₂O₃ modified Cr₂O₃ thick film is most sensitive to NH₃ gas at 100°C among all other tested gas.

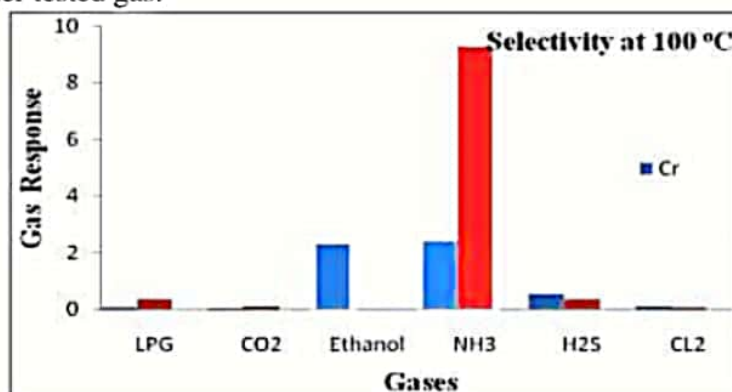


Fig. 6: Selectivity of pure and Fe₂O₃ modified Cr₂O₃ thick films.

Fig. 7 exhibits the relation between gas response of pure and Fe₂O₃ modified Cr₂O₃ thick films and concentration of NH₃ gas at 100°C. It is clear from the figure that the gas response of Fe₂O₃ modified Cr₂O₃ thick film (3 min dipping) increases linearly with gas concentration up to 100 ppm. The rate of increase in response was relatively larger up to 100 ppm and saturated beyond 100 ppm. The active region for the ammonia sensor is up to 100 ppm. At low gas concentration, the monolayer of NH₃ gas molecules formed on the surface of film. So, the NH₃ response increases in proportion up to active region. At high gas concentration, the multilayer of NH₃ gas molecules on the sensor surface would result into saturation in response beyond 100 ppm gas. So, for proper functioning, the sensor should work in the active region only.

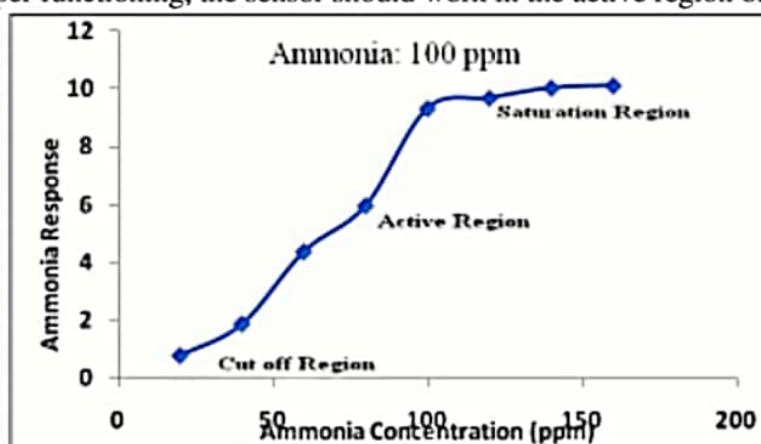


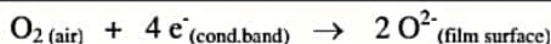
Fig 7: Variation of NH₃ response with NH₃ concentration (ppm)

The response and recovery of the Fe₂O₃ modified Cr₂O₃ thick film (3 min. dipped) to 100 ppm of NH₃ are 7 s and 10 s respectively. Thus the sensor showed very rapid response and recovery time to NH₃ gas. For better performance of the sensor the recovery should be very fast. When the gas exposure was switched off, the sensor returned back to its original chemical status, within very short time (10 s). This is the main feature of this sensor.

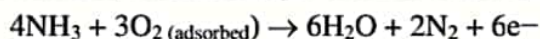
The response of this sensor towards 100 ppm of NH₃ gas at 100°C was measured for two month in the interval of 10 days and found almost constant. The good stability could be attributed to calcinations at high temperature and aging of sample for a few days. Thus the sensor showed a very stable response confirming the stability and hence reproducibility of the material.

Gas sensing mechanism

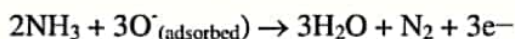
In sensor, the surface is the region where periodicity of the crystal can be interrupted. Due to this, localized energy levels are formed in the forbidden gap, which can either capture electrons or give up electrons. In case of pure or Fe₂O₃ modified Cr₂O₃, the surface oxygen ions donate electrons. The response could be attributed to the adsorption-desorption type sensing mechanism. Gas Oxidation depends upon the amount of oxygen species ($O_2 \rightarrow 2O \cdot \rightarrow O^{2-}$) adsorbed on the surface. Gas sensing mechanism is mostly explained in terms of conductance, by adsorption of atmospheric oxygen on the surface or by direct reaction of lattice oxygen or interstitial oxygen with the test gases. The working principle of thick film semiconducting gas sensors is based on the change of the electronic conductivity of the semiconducting material on exposure of target gas. In such mechanism, the atmospheric oxygen molecules O₂ (air) are adsorbed on the surface of the thick film. They capture the electrons from the conduction band of the thick film material as:



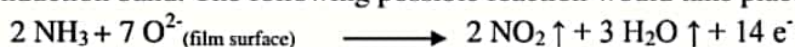
It would result in decreasing conductivity of the film. As we know that, the lone pair of electrons of NH_3 provides strong electron acceptor behaviour. But it acts as an electron donor to the metal oxide, when reacted with the adsorbed oxygen ions on the surface by returning the trapped electrons to conduction band. The reactions that generates free electrons when the number of oxygen ions reacted with NH_3 molecules, given in the following equations [21].



Or



Upon exposure, ammonia molecules react with adsorbed oxygen on the surface of the film got oxidized to nitrogen oxide gas and water vapors as the products liberating free electrons in the conduction band. The following possible reaction would take place.



Conclusions

The results of pure and Fe_2O_3 modified Cr_2O_3 thick films can be summarized as follows

1. The pure Cr_2O_3 film was more conductive than Fe_2O_3 modified Cr_2O_3 thick film (3min dipped).
2. Fe_2O_3 was found to be outstanding in enhancing the gas sensing performance of Cr_2O_3 based gas sensors.
3. Surface modification by dipping process was one of the most useful methods of modifying the thick film surface for the gas sensing performance of metal oxide based gas sensors.
4. A pure Cr_2O_3 thick film was almost insensitive to all tested reducing gases.
5. Fe_2O_3 modified Cr_2O_3 thick film (3 min dip) showed higher gas response to 100 ppm at 100°C .
6. The sensor showed good selectivity to NH_3 gas against Cl_2 , LPG, CO_2 , H_2S and $\text{C}_2\text{H}_5\text{OH}$ gases at 100°C .
7. The sensor showed very rapid response and recovery time to NH_3 gas.

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