

**LITERATURE SURVEY AND SCOPE OF THE
PRESENT INVESTIGATION**

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In this chapter, a literature survey on the synthesis and characterization of niobium based metal oxides and their gas sensor applications are presented in detail. At the end of this chapter, scope of the present investigation is given.

2.1. INTRODUCTION

Niobium is an important element in catalysis and its properties have been extensively analyzed [1, 2]. Ziolk and Nowak reviewed the structures, characterization and applications of niobium compounds [3]. Niobium pentoxide (Nb_2O_5) is a highly effective oxide support material for metallic catalysts. There are a significant number of niobium-containing materials (i.e. NbCl_5 , NbF_5 , NbH , NbS_2 , NbN , NbC , NbO_x and Nb organometallic compounds) that find catalytic applications [1].

2.2. LITERATURE SURVEY

T. A. Jones et al [4] carried out the electrical behaviour of some ceramic oxides at 900 °C as a function of oxygen pressure. Measurements have been made in an inert (N_2) atmosphere and in an inert atmosphere containing reducing gases (CO , H_2 and CH_4). The conductivities of Cr_2O_3 , Nb_2O_5 and CeO_2 showed a switch-like behaviour at the stoichiometric mixture of oxygen and reducing gas. Ga_2O_3 and ThO_2 and do not

exhibit this type of behaviour, the conductivity changing monotonically with increasing oxygen pressure.

C. Cantalini et al [5] reported the NO, NO₂ and NO_x gas-sensitivity properties of Nb-doped α-Fe₂O₃ sintered compact in 0–100 ppm of gas concentrations with 150–300 °C temperature ranges, by d.c. and a.c. techniques.

Jianzhong Zhu et al [6] prepared TiO₂-doped Nb₂O₅ thin film using the ion-beam enhanced deposition technique and the development of a new automobile air/fuel sensor with an additionally integrated platinum temperature sensor and a Pt heater.

S.Geoff et al [7] studied the response of gas-sensitive resistors fabricated from solid solution compounds of the form (CrNbO₄)_x(Sn_{1-y}Sb_yO₂)_{1-x} (0 < x < 1; y = 0, 0.01, 0.05) to carbon monoxide, propane and water have been studied.

D. Rosenfeld et al [8] investigated the electrical properties of thin-film metal-oxide-metal Nb₂O₅ oxygen sensors in the temperature range 400–600°C.

Rajnish K. Sharma et al [9] studied the electrical conductivity of TiO₂ material is on the operating temperature and oxygen partial pressure. This characteristic of the material has been exploited for use as an oxygen gas sensor.

L.Chambon et al [10] the sensitivity of Nb₂O₅ to NH₃ is studied, and NH₃ behaved like an electron donor and induced an increase of the n-type semiconductor Nb₂O₅ conductivity. In presence of oxygen, the conductivity decreased and the sensitivity of the Nb₂O₅ oxide to the NH₃ gas was much lower than in absence of oxygen. Humidity injects electronic carriers in the Nb₂O₅ material and acts like an electron donor. In presence of humidity, the conductivity of the Nb₂O₅ layer to NH₃ is improved.

Katarzyna Zakrzewska et al [11] the effect of Nb and Cr dopants as well as Sn⁴⁺ additions on the electronic structure of rf-sputtered TiO₂ thin films and its subsequent influence on gas sensor performance is reported.

D. Richter et al [12] reported development and characterization of a compact mid-infrared source for high-resolution spectroscopic detection of trace gases such as methane and water vapour at 3.3 μm in ambient air. This source utilized difference frequency generation (DFG) in a periodically poled LiNbO₃ (PPLN) crystal pumped by two single-frequency diode lasers. A maximum DFG power of 1.6 μW at 3.6 μm was generated with a pump power of 61.4 mW at 832 nm and a signal power of 41.5 mW at 1083 nm incident on a 19-mm long PPLN crystal, which corresponds to a conversion efficiency of 335 μW W⁻² cm⁻¹.

M. Z. Atashbar et al [13] prepared titanium dioxide (TiO₂) thin films using the sol-gel method and subsequently doped with niobium oxide (Nb₂O₅) for use in oxygen sensing applications.

Mary E et al [14] synthesised pulsed laser deposition (PLD) of a mesoporous niobium oxide film, and characterized by FT-IR spectroscopy, X-ray diffraction, scanning electron microscopy and transmission electron microscopy and used as dielectric phase in capacitive-type chemical sensors.

M. C. Carotta et al [15] fabricated thick films of nanostructured TiO₂ and Niobium-doped TiO₂ by screen-printing technology starting from pure Titania and Niobium-doped Titania powders; the powders were prepared by laser pyrolysis method which provides nanosized particles. This TiO₂-based thick film sensors exhibit a suitable sensitivity to atmospheric environmental monitoring.

A. Kohli et al [16] investigated Niobium pentoxide (Nb_2O_5) as a lean-burn oxygen sensor in the 400–800°C temperature range. It showed excellent sensitivity and good reversibility upon exposure to increasing and decreasing oxygen partial pressures. In the 10 ppm to 100% oxygen partial pressure range, the response of Nb_2O_5 sensor was as fast as the YSZ sensor. There is a change in the resistance when Nb_2O_5 is exposed to CO, CH_4 and CO_2 .

K.I.Gnanasekar et al [17] reported preparation, electrical, magnetic and gas sensor characterization of monoclinic phase (wolframite) of FeNbO_4 . Sensor studies on FeNbO_4 at various operating temperatures shows that the material is highly sensitive to H_2 and H_2S at 573-623 K. A moderate sensitivity for LPG was obtained at around 673 K.

E. Comini et al [18] reported possibility to improve the sensitivity towards ethanol and methanol of nanocrystalline TiO_2 thin films by doping with Nb and Pt. The thin films were prepared using the sol–gel process by the spin coating technique on alumina substrates. The relative change of the conductance of the thin film due to the introduction of 500 ppm of ethanol was as high at an operating temperature of 300°C, making them feasible for development of breath analysers.

M. Ferroni et al [19] Pure and Nb-doped TiO_2 thick-films were prepared by screen-printing It has been shown that niobium addition inhibited grain coarsening and hindered anatase-to-rutile phase transition. These semiconducting films exhibited n-type behavior, while Nb acted as donor–dopant. Gas measurements demonstrated that the films were suitable for CO or NO_2 sensing.

Okamoto koji et al [20] a compact chlorine gas sensor was fabricated by using monovalent Cl^- anion conducting calcium-doped lanthanum oxychloride and trivalent

Al³⁺ cation conducting (Al_xZr_{1-x})₄(4-x)Nb(PO₄)₃(x=0.2). The response time necessary to attain a 90% response within several min and reproducible enough for a practical application.

Weber et al [21] reported Nanometric SnO₂·Nb₂O₅ powders with the purpose of developing an ethanol sensor. The powders were prepared by the Pechini method, morphologically characterised by X-ray diffraction, and their specific surface area determined by BET, after which they were subjected to ethanol vapour sensitivity tests. A correlation was established between the microstructure of the material, the effects of the dopant and the sensors response.

G. Song et al [22] investigated the static and kinetic properties upon hydrogen absorption desorption in epitaxial niobium films by in situ and time-resolved resistivity measurements.

Ana Ruiz et al [23] prepared nanocrystalline TiO₂ modified with by sol-gel technique and used as a sensor material for CO and ethanol.

A. Ruiz et al [24] reported Nb-doped TiO₂ nanopowders. The niobium introduction retarded the anatase-to-rutile transformation and hinders grain growth mechanisms. The electrical behaviour studied for CO and ethanol gases showed that the response to ethanol was slightly diminished by the incorporation of Nb atoms and not affected by the structure modification. However, the response to CO was modified by structure changes such as the anatase/rutile transformation, grain size and Nb segregation. For the samples treated at 700 °C, the best response to CO was achieved for 4 at.% of Nb.

Huankiat et al [25] the electrical and defect properties of langasite (La₃Ga₅SiO₁₄) were studied as a function of temperature, oxygen partial pressure and dopants in order

to characterize its electrical behavior in relation to its performance as a bulk acoustic wave (BAW) gas sensor operating at elevated temperatures. Undoped, 5%–Nb donor doped, and 1%–Sr acceptor doped langasite specimens were studied.

Ming-Cheng Wu et al [26] developed a calorimetric sensor utilizing a thermoelectric device supported on a planar alumina substrate. By using a highly selective carbon monoxide (CO) oxidation catalyst and a non-selective platinum (Pt) catalyst, the device can be built to detect either CO or hydrocarbons with high selectivity. The CO oxidation catalyst comprised of lead-modified platinum and exhibited excellent selectivity at 200–400 °C. The thermoelectric device consisted of two thick film junctions made of niobium pentoxide (Nb₂O₅)-doped titanium dioxide (TiO₂) and a lithiated nickel (Ni), which were supported on a planar alumina substrate. The thermocouple detected the difference in temperature due to different catalytic reactions over the two junctions and showed a high output signal because of the high Seebeck coefficient of Nb₂O₅-doped TiO₂ (–400 μV/°C). In gas bench tests, the sensor had a linear output of 0–2.75 mV over 0–1000 ppm of propylene and a response time of 2.5 s (at 90% of amplitude) at a gas temperature of 350 °C.

Isao Hasegawa et al [27] reported a new solid electrolyte type nitrogen monoxide (NO) gas sensor which can operate in the intermediate temperature region. This was fabricated by the combination of trivalent aluminum cation conducting (Al_{0.2}Zr_{0.8})_{20/19}Nb(PO₄)₃ and divalent oxide anion conducting yttria stabilized zirconia (YSZ) with LiNO₃-doped (Gd_{0.9}La_{0.1})₂O₃ as the sensing auxiliary electrode.

Ana M. Ruiz et al [28] reported nanocrystalline titanium dioxide after hydrothermal treatment was loaded singly or doubly with various metals or metal oxides

(Au, Ag, Pt, Pd, Cu, Co, Nb, V). Hydrothermally treated TiO₂ exhibited enhanced thermal stability, resistance to grain growth and phase transformation. Incorporation of metal additives further decreased the grain size of titania, while the phase transition was generally promoted, except in the case of Au and Pd, which maintained predominant anatase phase at 800 °C. Among the different additives tested, Au was found to be the most attractive promoter for the CO sensing properties. Therefore, studies were extended to several Au concentrations on TiO₂ and in addition, to simultaneous loads of Au and Nb or V. Doubly modified Au– and Nb–TiO₂ presented the highest Au content on surface, as inferred from XPS measurements, and exhibited the largest and fastest sensor response to CO at high operating temperatures.

E. Comini et al [29] prepared thin films of titania with the addition of niobium and tantalum by reactive sputtering process. The films proved sensitive to ethanol and carbon monoxide and ammonia. In the case of niobium addition, it was shown that annealing temperature and niobium content strongly influenced the gas response of the films converting a n-type response.

T. Anukunprasert et al [30] reported titanium dioxide (TiO₂) gas sensor for combustion and exhaust air pollutants monitoring. The strongly depended its properties such as thermal stability, grain size and surface area. In this study, nanostructure TiO₂ with its thermal stability enhanced by niobium dopant (Nb–TiO₂). Nb-doped TiO₂ at a level of 3–5 mole% clearly hindered the anatase to rutile phase transformation and inhibited the grain growth in comparison with pure TiO₂. The sensitivity of CO was significantly increased with an increase of thermal stability of Nb-doped TiO₂ in

comparison with those of undoped TiO_2 and thus is useful for CO sensing studies at high temperatures.

Takeo Hyodo et al [31] investigated H_2 sensing properties of an anodically oxidized Nb_2O_5 film coupled with a noble metal electrode (M/ Nb_2O_5 , M: Au, Pt and Pd) under various operating conditions. Among the sensors tested, Pd/ Nb_2O_5 showed the highest H_2 response, and the logarithmic sensor current under a forward bias was proportional to the logarithmic H_2 concentration in the whole range tested (10–8000 ppm). The current–voltage characteristics of Pd/ Nb_2O_5 (S) sensor at 100 °C apparently showed a typical rectifying function of a metal–semiconductor junction, which was formed between the Pd electrode and the Nb_2O_5 thin film.

Miguel Adolfo Ponce et al [32] prepared thick films with undoped nanometric SnO_2 particles and Co, Nb, Fe-doped SnO_2 for the purpose of developing oxygen and carbon monoxide gas sensors. It was determined that the SnO_2 -doped films have a greater sensitivity between 200 °C and 350 °C.

A. Ponzoni et al [33] reported the synthesis and characterization of SnO_2 films with varying Nb and V content for sarin detection.

E. Sotter et al [34] prepared pure titania and Nb-doped titania based sensors synthesised by sol–gel method and calcined at temperatures between 600 °C and 900 °C. A comparison of the response to traces of O_2 in N_2 balance of the different samples was reported at working temperatures between 300 °C and 600 °C.

K. Arshak et al [35] prepared thick film capacitors with an oxide dielectric layer using niobium pentoxide (Nb_2O_5), titanium dioxide (TiO_2) and cerium dioxide (CeO_2). SEM was used to examine the morphology and particle size of the thick films. To aid in

the development of a suitable interface circuitry, the frequency and temperature dependence of each device was investigated. Each sensor was then subjected to pressures in the region of 0–100 kPa. The results showed that capacitors with an Nb₂O₅ dielectric layer were most sensitive.

Soumya Kanti Biswas et al [36] synthesised nanosized iron niobate (FeNbO₄) powder from precursor solution containing iron nitrate, niobium tartarate, and triethanolamine (TEA). The compressed powder exhibited maximum sensitivity to H₂ at 250°C and liquefied petroleum gas (LPG) and NH₃ at 300°C. The incorporation of platinum (1 wt %) into the nanosized FeNbO₄ by impregnation method reduced the operating temperature along with response time and recovery time for sensing H₂, LPG, and NH₃.

A.Z. Sadek et al [37] reported polyaniline/WO₃ nanofiber composite-based surface acoustic wave (SAW) gas sensor towards hydrogen (H₂). Chemical oxidative polymerization of aniline was employed to synthesize polyaniline nanofibers with WO₃ nanoparticles. The sensor was exposed to various concentrations of H₂ gas and operated at room temperature. The sensor response was found to be 7 kHz towards 1% of H₂ in synthetic air. A fast response and recovery in a stable baseline condition were observed at room temperature.

Nguyen Van Hieu et al [38] prepared Nb-Pt co-doped TiO₂ and the hybrid SWCNTs/Nb-Pt co-doped TiO₂ thin films by the sol–gel spin-coating process for gas-sensor fabrication. Experimental results revealed that the responses to ethanol of Nb–Pt co-doped TiO₂ sensors with SWNCTs inclusion increase by factors of 2–5 depending on

the operating temperature and the ethanol concentration, compared to that of the sensor without SWCNTs inclusion.

Alexandra Teleki et al [39] prepared Nb- and Cu-doped TiO₂ nanoparticles by flame spray pyrolysis (FSP) and tested for sensing of CO and ethanol at 400 °C in dry air.

Soumya Kanti Biswas et al [40] synthesised nanosized CuNb₂O₆ by thermal decomposition of an aqueous precursor solution containing copper nitrate, niobium tartrate and tri-ethanol amine (TEA), followed by calcination at 700 °C for 2 h. Appreciable changes in resistance of polycrystalline nanosized CuNb₂O₆ upon exposure to reducing gases like hydrogen, liquefied petroleum gas (LPG) and ammonia in ambient atmosphere recognize the material as a gas sensor.

E Llobet et al [41] prepared hybrid titania films using sol–gel method for obtaining well-dispersed hydrogen plasma-treated multiwall carbon nanotubes in either pure titania or Nb-doped titania. The gas sensitivity studies performed on the different samples had shown that the hybrid layers based on titania and carbon nanotubes possessed an unprecedented responsiveness towards oxygen.

Takeo Hyodo et al [42] prepared a thermally oxidized TiO₂ or Nb₂O₅ film equipped with a top Pd film electrode and a bottom Ti or Nb plate electrode has been investigated as a diode-type H₂ sensor under air or N₂ atmosphere. Pd/TiO₂(n)/Ti sensors showed relatively poor H₂ sensing properties in air, in comparison with Pd/anodic-TiO₂(n)/Ti sensors constructed with an anodized TiO₂ film equipped with a top Pd film electrode and a bottom Ti plate electrode, which were reported in their previous studies. On the other hand, Pd/Nb₂O₅ (n)/Nb sensor showed relatively larger

H₂ response with fast response and recovery speeds than Pd/TiO₂ (n)/Ti sensor in air under high forward bias conditions.

Michao Zhang et al [43] synthesized perovskite niobate (BaNbO₃) nanocubes by using the composite-hydroxide-mediated method. The responsiveness to humidity for static and dynamic testing proved the ultrasensitive properties of the sensors. The resistance changed from 932.4 MΩ to 2.2 MΩ as the relative humidity (RH) increased from 10% to 80%. The response-time and recovery-time of the resistance was 12 s and 32 s versus the changes of relative humidity from 20% to 60%. These results indicated promising applications of BaNbO₃ nanocubes in a highly sensitive environmental monitoring and humidity controlled electronic device.

C.G. Dighavkar et al [44] prepared pure and Nb₂O₅ doped TiO₂ thick films of various concentrations (1 wt.%, 3 wt.%, 5 wt.%, 7 wt.% and 10 wt.%) on alumina substrate by using screen printing technique. These films were then fired at temperature 800 °C for two hours in O₂ atmosphere. Morphological, compositional and structural properties of the samples were performed by means of several techniques, including scanning electron microscopy (SEM), energy dispersive spectroscopy (EDS), X-ray diffraction techniques. H₂S gas sensing phenomena observed in Nb₂O₅ doped-TiO₂ screen-printed thick films. The surface resistance of thick films decreased when exposed to H₂S gas. Low concentration of doping in Titania (TiO₂) with Nb₂O₅ has shown significant enhancement in sensitivity (98.78 %) to H₂S gas than pure TiO₂ film at 200 °C with fast response and recovery time.

Laure Chevallier et al [45] three Pt|YSZ| metaloxide (MOx) planar electrochemical cells were prepared, based on commercial tape-cast YSZ layers, using Nb₂O₅, Ta₂O₅, and a

$\text{Nb}_2\text{O}_5:\text{Ta}_2\text{O}_5$ mixture in 1:1 weight ratio as MO_x sensing electrodes, and their sensing performance was compared. All the sensors showed outstanding responses to 1000 ppm of propene in air and propene sensitivities at 700°C , being -214 mV and -190 mV/dec, respectively, the largest for Ta_2O_5 electrode-based sensors. However, the sensors using the $\text{Nb}_2\text{O}_5:\text{Ta}_2\text{O}_5$ mixture electrode showed the best performance in terms of compromising selectivity and stability with keeping large response and sensitivity values.

Sukon Phanichphant et al [46] Undoped TiO_2 and TiO_2 nanoparticles doped with 1–5 at.% Nb were successfully produced in a single step by flame spray pyrolysis (FSP). The phase and crystallite size were analyzed by XRD. The BET surface area of the nanoparticles was measured by nitrogen adsorption. The trend of SSABET on the doping samples increased and the BET equivalent particle diameter (dBET) (rutile) increased with the higher Nb-doping concentrations while dBET (anatase) remained the same. The morphology and accurate size of the primary particles were further investigated by high-resolution transmission electron microscopy (HRTEM). The crystallite sizes of undoped and Nb-doped TiO_2 spherical were in the range of 10–20 nm. The sensing films were prepared by spin coating technique. The mixing sample was spin-coated onto the Al_2O_3 substrates interdigitated with Au electrodes. The gas sensing of acetone (25–400 ppm) was studied at operating temperatures ranging from $300\text{--}400^\circ\text{C}$ in dry air, while the gas sensing of ethanol (50–1,000 ppm) was studied at operating temperatures ranging from $250\text{--}400^\circ\text{C}$ in dry air.

Naji Al Dahoudi [47] reported the synthesis of niobium doped titanium oxide (TiO_2) colloid was synthesized to fabricate a hydrogen gas sensor layer on oxidized silicon

wafer substrate. The layers were obtained using spin coating technique and then heated in air at 500°C for 30 min. The doping of TiO₂ led to a significant enhancement of the sensitivity of the layer especially at low operating temperature.

Viruntachar Kruefu et al [48] developed unloaded ZnO and Nb/ZnO nanoparticles containing 0.25, 0.5 and 1 mol% Nb were produced in a single step by flame-spray pyrolysis (FSP) technique. Nb/ZnO nanoparticles paste composed of ethyl cellulose and terpineol as binder and solvent, respectively was coated on Al₂O₃ substrate with gold electrodes to form thick films by spin coating technique. The influence on a low dynamic range of Nb concentration on NO₂ response (0.1 - 4 ppm) of thick film sensor elements was studied at the operating temperature ranging from 250 - 350 °C in presence of dry air.

Hi Gyu Moon et al [49] reported a novel route for the fabrication of highly sensitive and rapidly responding Nb₂O₅-based thin film gas sensors. The gas sensitivity of Nb₂O₅ films was enhanced through both the TiO₂ doping and the surface embossing. Upon exposure of 50 ppm of CO at 350 °C, a gas sensor based on TiO₂-doped Nb₂O₅ film exhibited a very high sensitivity of 75% change in resistance and a rapid response time of 8 s under 3 V, whereas a sensor based on plain Nb₂O₅ film showed a 70% resistance change and a response time of 65 s under 1 V.

I. Hasegawa et al [50] fabricated a new type of nitrogen monoxide (NO) gas sensor such as (Al_{0.2}Zr_{0.8})_{20/19}Nb(PO₄)₃ and yttria stabilized zirconia (YSZ) and (Gd_{0.9}La_{0.1})₂O₃ and its NO sensing performance was investigated. The time to attain 90 % response was less than two minutes for NO in concentration range of 200–2000 ppm.

2.3. SCOPE OF THE PRESENT INVESTIGATION

Gas sensors based on semiconductor metal oxides focused numerous research efforts during last few years. Among them mesoporous Nb_2O_5 has taken much attention because of its special properties and its interesting applications, such as electrochromic, photocatalytic hydrogen production, gas sensor, and also electrode material in electro catalysis, porous coating in electrochemical solar cells etc. Since sensor made of these oxides can exhibit sensitivity for a host of gases. However, efforts are being made to enhance the selectivity and sensitivity of given material towards a particular analyte gas. One approach to achieve this goal is through surface modification by proper choice of additives or dopants to the base oxide materials. .Towards this end, transition metals have been used as successful doping materials for Nb_2O_5 . It was observed that the resistivity of Nb_2O_5 is increased due to the depressed carrier concentrations which resulted in increase of electrical resistivity of the metal oxide, such high resistivity effect has been shown to enhance the sensitivity of the metal oxide based gas sensors. In addition, the possibility of shifting the base material of sensor to a completely different oxide system is also considered. Recently, some niobium based binary oxides such as InNbO_4 , CrNbO_4 , CeNbO_4 and LaNbO_4 are being prepared by solid state reaction method. It is known that the preparation method and choice of metal precursor have an important effect on the dispersion, specific surface area, morphology, and other structural and textural properties of metal oxide which influence the sensor performance. The sensing mechanism is also based on the surface reaction of the particles with the exposed gas. The nanosized semiconducting metal oxides are desirable for gas sensor applications. Hence, in the present investigation,

niobate based sensor materials such as InNbO_4 , CrNbO_4 , AlNbO_4 , LaNbO_4 and FeNbO_4 by niobium-citrate complex process and their physical characterization and sensor application for LPG, NH_3 and ethanol are studied as follows;

- ❖ The thermal analysis (TG/DTA) will be used to understand the thermochemical properties such as decomposition temperature and phase formation temperature etc., of the prepared precursor samples.
- ❖ The as prepared powder will be calcined at optimized temperature and the phase purity and structure of the resultant powders will be confirmed by X-ray diffraction analysis.
- ❖ The morphology of prepared nanopowders will be observed using scanning electron microscope (SEM) studies.
- ❖ The particle size and crystalline quality of the prepared nanopowders will be confirmed by transmission electron microscope (TEM) studies.
- ❖ Chemical composition of the prepared nanopowder will be analyzed using energy dispersive X-ray spectroscopy (EDX) studies.
- ❖ The optical band gap energy of the prepared nanopowders will be measured using UV-Vis absorption spectra, equipped with diffuse reflectance spectroscopy (DRS).
- ❖ The surface area of prepared nanopowders will be measured using the BET nitrogen adsorption and desorption studies.
- ❖ The conductivity of the prepared nanopowder will be studied as a function of temperatures using the impedance studies.

- ❖ Finally, the gas sensing behaviour of the prepared nanopowders will be studied for gases like Liquid petroleum gas (LPG), ammonia (NH_3), and ethanol ($\text{C}_2\text{H}_5\text{OH}$) as a function of various controlling factors like operating temperatures, gas concentrations and response time.

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