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Experimental study of the cobalt oxide etching rate from metal surface using F2/He atmospheric pressure plasma for decontamination of nuclear components

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HIGHLIGHTS

- Metal surface cleaning or etching techniques using reactive cold plasma as emerging technique is presented.
- Removing Cobalt oxide from metal surface with presenting removal rate has been done.
- Scanning electron microscopy with energy dispersive X-ray spectroscopy and the purification rate was performed.

ABSTRACT

Metal surface cleaning or etching techniques using reactive plasma are emerging as one of the dry processing techniques for surface contaminants with high bond energy, especially for cleaning and decontamination of nuclear components and equipment. In this study, the plasma reaction due to the discharge of a dielectric barrier of a mixture of 95% helium and 5% fluorine with cobalt oxide film (Co_3O_4) grown on the surface of stainless steel 304 was studied experimentally. Experimental results show that cobalt oxide becomes a powder after plasma irradiation and is easily separated from the surface of the base metal. The optimal plasma generating conditions of the dielectric barrier discharge used in this experimental study were obtained at atmospheric pressure, voltage 4.5 kV, and frequency 25 kHz with an etching rate of 10.875 μmol.min^{-1}. The samples were analyzed before and after plasma irradiation, using Scanning Electron Microscopy with Energy Dispersive X-ray spectroscopy and the purification rate was performed using a sequential weighting of the samples with scales 10^{-4} g accurately obtained. The results show the ability of this method to effectively remove the surface contamination of cobalt from the surface of stainless steel 304.

KEYWORDS

Metal waste decontamination
Plasma surface cleaning
Etching rate
Cobalt oxide
Dielectric barrier discharge

HISTORY

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1 Introduction

Under continuous energy utilization, many key components and equipment, especially in the primary circuit of nuclear power plants, are gradually contaminated by the adsorption of radioactive isotopes. If these surface contaminants can be selectively removed, the radioactive substrate metal can be converted to a non-radioactive or low-level radioactive material, which leads to a very large reduction in the amount of radioactive waste and significant economic benefits if the substrate is recycled. The OECD/NEA (Organisation for Economic Co-operation and Development/Nuclear Energy Agency) has recently claimed that approximately 30 million tons of contaminated scrap metal will be generated over the next 50 years from the decomposition and decommissioning of nuclear facilities (Chen, 1997). The International Atomic Energy Agency predicts that 650 tons of radioactive metal waste will be generated due to the deactivation of PWR with a capacity of 900 to 1300 MW (Coates et al., 2008).

Current cleaning processes such as wet chemical processes, mechanical machining and, gas phase cleaning processes with high reliability have disadvantages in producing large amounts of secondary waste. A dry process such as a plasma vacuum process may avoid the secondary waste problem, but the process volume, in this case, is very

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limited because the plasma processing volume is limited by the size of the plasma vacuum chamber. To overcome these shortcomings, the plasma cleaning method, which works at atmospheric pressure, has emerged as a new and promising technology (Kim et al., 1999; Nam and Um, 2022; Lee et al., 2022; Veilleux et al., 2000). Techniques for cleaning or etching metal surfaces using reactive plasma gas are emerging as one of the dry processing techniques which are for surface contaminants with high bond energy, especially used to clean used or used core parts and equipment (Kim et al., 1999; Nam and Um, 2022; Kim et al., 2004, 2003a,b; Windarto et al., 2000a). This method can maintain the need for minimal secondary waste production while maintaining the same efficiency level of wet cleaning techniques (Kim et al., 2004; IAEA, 1999). In principle, this method selectively selects surface contaminants, converts them to volatile compounds through the catalytic surface, and finally removes them from the surface (Kim et al., 1999; Nam and Um, 2022; Kim et al., 2003a,b; Windarto et al., 2000a; Mehrabifard, 2023; Amakawa, 2001; Toumanov, 2003; Girold and Barthelemy, 2001; Martz, 1991).

Co-58 and Co-60 are the most troublesome radioactive nucleotides that are cleared in many cases, including the Inconel vapor tubes shown in Fig. 1 (Kim et al., 2004). As a result, the removal of Co isotopes from the contaminated metal surface is one of the main strategies in the development of the metal surface cleaning process.

Several research groups have investigated the effectiveness of plasma etching for decontamination purposes. Tatenuma et al. reported in (Tatenuma et al., 1998) that chemically reactive plasma treatment using non-toxic gases is a viable method for gas-phase decontamination, as it provides mild conditions such as reduced pressure, shorter treatment durations, and ambient temperature. Kim et al. (Kim et al., 1999) also demonstrated the ability of CF_4/O_2 plasma to convert UO_2 to UF_6 in a two-step process, facilitating the handling of radioactive materials.

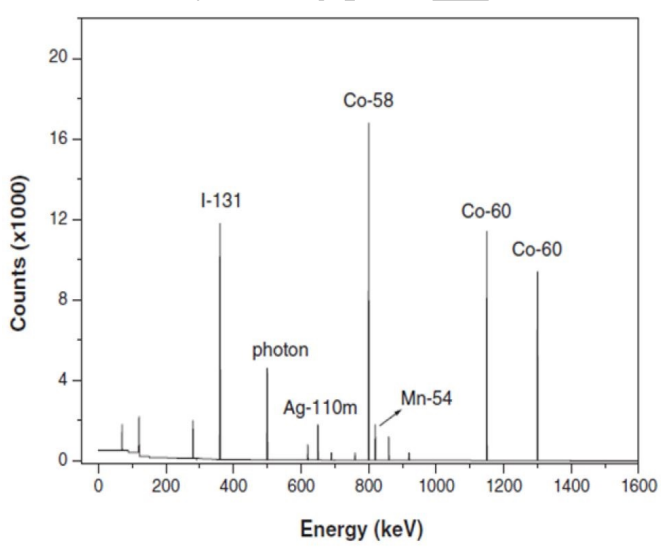

Figure 1: Gamma-ray spectroscopy of an Inconel alloy tube taken out of a steam generator belonging to an old nuclear reactor (Kim et al., 2004).

Windarto et al. (Windarto et al., 2000a) showed the effectiveness of a microwave atmospheric pressure plasma jet in cleaning radioactive materials for the first time. They utilized a CF_4-O_2-based microwave discharge to decontaminate radioactive CoO_2 from a stainless steel surface at 1.5 kW. Kim et al. (Kim et al., 2003a,b) conducted fluorination reactions of UO_2 and surface etching of metallic Cobalt surfaces with a CF_4/O_2 mixed gas plasma, achieving high reaction rates even at elevated temperatures.

Yong-Hwan Kim et al. (Kim et al., 2004) developed an atmospheric pressure plasma torch source for decontaminating Cobalt-contained oxide layers on metal surfaces, utilizing helium-based discharge and Argon-based discharge with O_2 and CF_4 as reactive gases. However, achieving stable plasmas with such reactive gases at atmospheric pressures was challenging. Yang et al. (Yang et al., 2004) used similar plasma composition (CF_4/O_2/He) to discharge UO_2 from a stainless-steel surface, leading to promising results for transuranic waste decontamination.

Suzuki and his colleagues compared different methods of removing radioactive contamination with plasma and evaluated atmospheric pressure methods as a suitable method for cleaning wide surfaces such as inside the reactor vessel. They used a gas composition of 70% He, 24% CF_4, and 6% O_2 to remove Cobalt oxide pollution (Suzuki et al., 2005). NF_3 gas has been found to be an efficient etchant gas in plasma processing, particularly for Cobalt and its oxides, due to its higher fluorine dissociation rates compared to CF_4 and SF_6 (Lee et al., 2019). Researchers examined the plasma etching reaction of Cobalt oxide film grown on Inconel base metals using NF3 gas to demonstrate the high decontamination rate of steam generator tubes contaminated with radioactive Co isotopes (Lee et al., 2019). Their latest work published in 2022 showed that plasma surface decontamination is highly effective in removing contaminated nuclides such as Cobalt attached to aggregate in concrete generated during the decommissioning of nuclear power plants (Lee et al., 2019). However, high enough plasma etching rates have not been achieved yet to practically decontaminate Cobalt (Kim et al., 2004) and Cobalt oxides (Kim et al., 2003b).

In this study, the main contamination observed is Cobalt (Co) on stainless steel 304. The decontamination process focuses on removing radioactive gamma-ray emitters like Co-60 from the inside surface of the reactor vessel and tubing of the steam generator. However, the fuel clad is contaminated by alpha-ray emitters such as uranium and plutonium. According to (MacDonald et al., 1996), the coolant temperature at the steam generator inlet ranges from 290 to 320 °C, while at the outlet, it falls between 267 to 292 °C. Additionally, the normal operating temperature of the vessel is reported to be 566.3 K (Chaudhry et al., 2014). Consequently, cobalt oxide forms on the steel surface at approximately 500 degrees Celsius. Plasma removes cobalt oxide contamination by fluoridating (F) the contaminant and converting it easily removable powder. Cobalt removal from metal surfaces can be accomplished by chemical reaction of the plasma and the production of any volatile compounds or powders that are easily separated. Detachable powders such as CoF_2 and

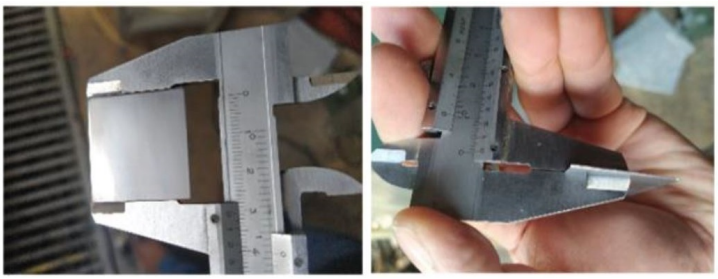

Figure 2: Samples made of stainless steel 304.

CoF_3 can be formed by fluorination in fluorine plasma. This plasma chemical reaction is shown in Eq. (1):

$$\text{Fluorination}: Co + F^* \rightarrow CoF_2, CoF_3 \ \ \text{solid} \qquad (1)$$

This important reaction can be strongly stimulated by the production of stable plasma discharges. Because fluorine radicals can be produced in basic helium-based plasma with some fluoride as the reactive gas (Kim et al., 2003b). Earlier works on the plasma fluorination reaction mechanism demonstrated that an intermediate species, a cobalt-fluorine-oxygen compound, i. e., $[CoF(ad).nO]^*$ formed on the surface and becomes equilibrated with surface atoms. Once the fluorine radicals from the gaseous plasma ($F_{(g)}$) successfully interact with the intermediate species (e.g., cobalt-fluorine-oxygen compounds), the newly formed reaction product, CoF_2, immediately desorbs from the surface. Then the kinetic scheme for this reaction can be described as follows in Eq. (2):

$$[CO_3O_4] \underset{k_a}{\overset{k_d}{\rightleftharpoons}} [CoF_{(ad)}.nO]^* \rightarrow k^* CoF_{2(g)} \qquad (2)$$

As wellknown, when $k^* \gg k_a$, the desorption rate becomes constant, i.e., $R_d = k_d[CoF_{(ad)}.nO]^*$. When this mechanistic understanding is taken into consideration, the overall reaction can be broken down into the following elementary Eqs. (3) and (5):

$$CO_3O_{4(s)} + F_{(g)} \rightarrow CoF_{(ad)}.nO^* \qquad (3)$$

$$CoF_{(ad)}.nO^* + F_{(g)} \rightarrow CoF_{2(g)} + nO \qquad (4)$$

R_d represents the etching rate of cobalt oxide, k_d is the rate constant for the desorption reaction, k_a is the rate constant for the adsorption reaction, k^* is the rate constant for the formation of intermediate species, and $[CoF_{(ad)}.nO]^*$ represents the concentration of adsorbed intermediate species on the surface of materials.

In this reaction, therefore, desorption of the reaction product, CoF_2, is the surface reaction rate limiting, following a linear kinetics law, although limited surface coverage of the intermediate species caused weight loss of the specimen to saturate as the reaction time progressed (Lee et al., 2022). Accordingly, the initial desorption rate is taken as the principal etching rate in this study.

Different methods for cobalt decontamination vary in terms of plasma etching apparatus, such as the plasma production source (RF, microwave, and kHz DBD) and apparatus geometry, as well as the type of gas used (CF_4, SF_6, NF_3, etc.), power, working frequency, and working pressure. The objective of all these studies is to achieve higher etching rates and greater efficiency (Kim et al., 2004; Lee et al., 2019; Suzuki et al., 2005; Windarto et al., 2000b). In the (Askari et al., 2022, ress) an atmospheric pressure DBD with CF_4 etching gas was simulated using Comsol software to determine optimal parameters in terms of dimensions, dielectric thickness, source power, and frequency.

The removal of cobalt from metal surfaces is a crucial aspect in developing surface cleaning methods for metals. Among various plasma generators, the DBD (dielectric barrier discharge) is selected for this study due to its ease of installation in steam generator tubes and the ability to utilize. Actually, DBD plasma is used for various applications (Mehrabifard, 2023; Omrani et al., 2023). While the microwave process exhibits fast etching speed, it consumes a significant amount of energy. On the other hand, the dielectric barrier process requires minimal power and can be considered a preferred choice. As for atmospheric pressure methods, they are suitable for cleaning large surfaces like inside reactor vessels. Therefore, the designed plasma apparatus in this research is based on atmospheric pressure. first, the method of sample making and cobalt oxide growth is explained and then by expressing the implemented experimental arrangement, the results obtained from the appropriate time to eliminate cobalt contamination and corrosion rate are presented.

2 Materials and Methods

2.1 Growth of cobalt oxide film on stainless steel 304 sample

According to Fig. 2, to prepare the cobalt oxide film on stainless steel 304, square pieces with dimensions (3×3 cm^2 and a thickness of 0.5 mm) were cut from a stainless steel 304 plate. The samples were polished with 320, 600, and 1200-micron grains of sandpaper and cleaned using a 50:50 solution C_2H_5OH:CH_3COCH_3 in an ultrasonic cleaner to produce a mirror-like surface. The samples were air-dried and then cobalt nitrate hexahydrate solution ($(Co\ (NO_3)_2\ 6H_2O)$ at a concentration of 300 $mg.ml^{-1}$ was applied to their surface. (300 microliters of the solution is poured on each sample) Then, the samples were baked in an electric oven at 500 °C for 2 hours to allow cobalt oxide films (as shown in Table 1) to grow on the base metal surfaces.

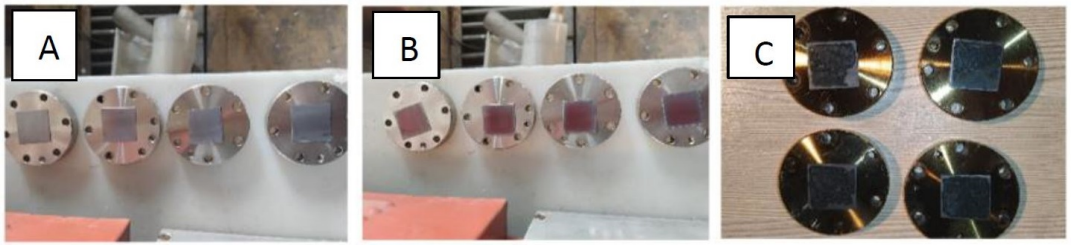

Figure 3: A, Samples before preparation. B, Samples before being placed in the furnace. C: Samples after leaving the furnace.

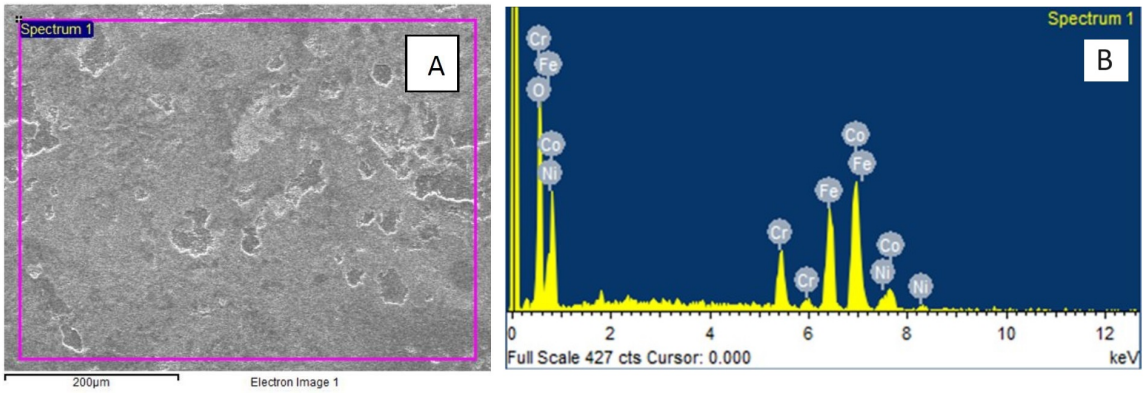

Figure 4: A: Image of the area where SEM/EDX analysis was performed after oxidation of cobalt on the sample. B: Graph of EDX analysis results.

Table 1: Specifications of cobalt oxide grown on steel samples (Lee et al., 2019).

Chemical formula	CO_3O_4
Molar mass	240.80 g.mol^{-1}
Density	6.11 g.cm^{-3}
Melting point	895 °C
Boiling point	900 °C

Table 2: EDX analysis, five elements have been identified.

Element	Weight (%)	Atomic (%)
O	26.40	56.13
Cr	8.08	5.29
Fe	23.82	14.51
Co	38.48	22.21
Ni	3.22	1.87

Figure 3 shows the samples before putting them in the furnace and after the oxidation of cobalt on the sample. After being removed from the furnace, the samples are cleaned by ultrasonic cleaning in a distilled water bath for 5 minutes to remove cobalt that has failed to form an oxide layer. To confirm the oxidation of cobalt on the sample, SEM imaging with EDX analysis was used, which is shown in Fig. 4. The images show the growth of cobalt oxide on the sample surface. In addition, according to EDX analysis, five elements have been identified, which are shown in Table 2.

2.2 Experimental Setup

A schematic diagram of the plasma etching device used in this study is shown in Fig. 5. Reaction gas in the amount of 300 SCCM (Standard Cubic Centimeters per Minute) is supplied to the 3.4-liter reaction chamber using the MFC (Mass Flow Controller) unit. A 13-liter capsule containing 95% helium gas and 5% fluorine gas was used and the power required to create an electrical discharge was applied by a 300 W neon transformer and its output was adjusted by an autotransformer between parallel electrodes. The frequency was fix and to achieve optimal performance, the power supply output voltage, distance between electrodes changed till to have stable and uniform discharges. The optimum plasma output voltage is 4.5 kV with a frequency of 25 kHz. The distance between the dielectric and the sample is 0.5 cm, also thermocouple was placed below the sample surface to measure the temperature of the sample. To create safe conditions in terms of fluoride gas activity, the gas capsule was placed in an open area and the complete reaction path was closed and any fluorine leakage into the laboratory environment was prevented. Direct evacuation of gas to the environment is prevented and to evacuate the gas, the gas is first introduced into NaOH solution so that fluorine gas is converted to HF acid by combining in this solution and is no longer released as dangerous gas in the environment. Despite the mentioned considerations, all operations have been performed with chemical masks.

2.3 Plasma etching tests

Before the main plasma etching tests, the entire chamber was filled with gas for 5 minutes to completely remove impurities and residual moisture from the surface of the chamber wall with the desired gas flow. The gas inlet flow rate was fixed at 300 SCCM and the total test

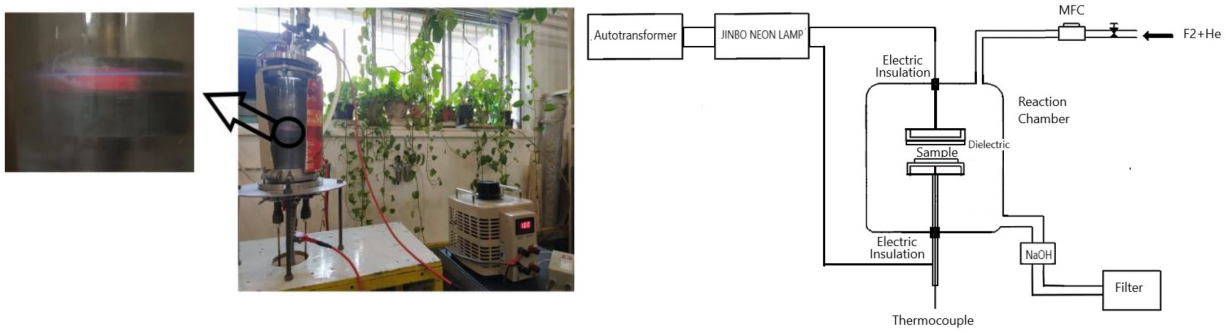

Figure 5: Schematic of the experiment.

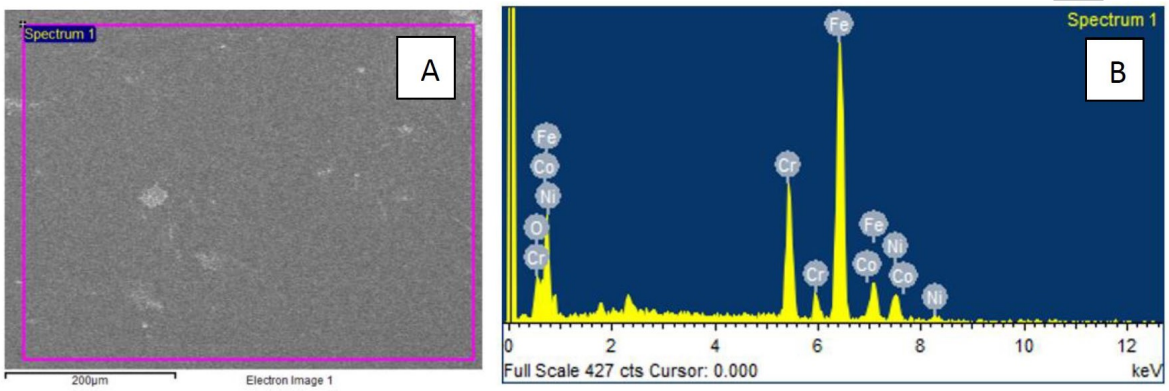

Figure 6: A: Image of the area where SEM/EDX analysis was performed after plasma irradiation. B: Graph of EDX analysis results.

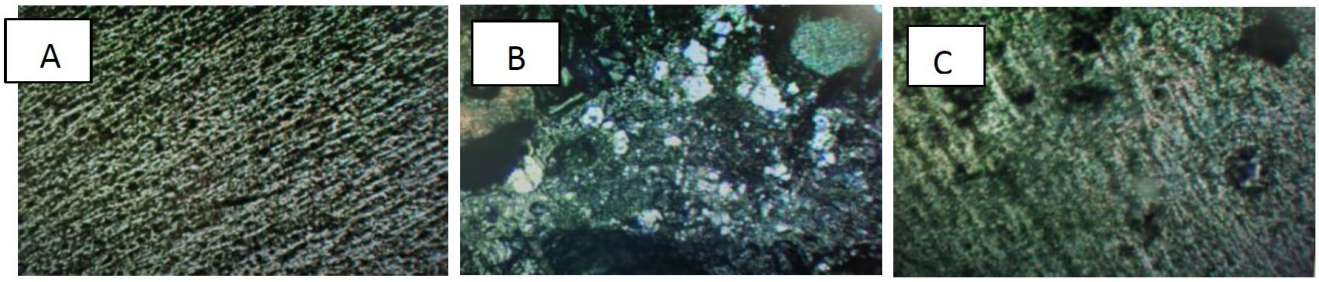

Figure 7: The OM images of the sample surface. A: Before cobalt is oxidized on the sample surface. B: After cobalt oxidation on the sample surface. C: After plasma irradiation.

time to reach the maximum decontamination percentage was about 8 minutes. In the experiments performed, the weight of each sample was measured every 2 minutes and the decontamination process was continued again.

The etching rate was determined using the weight loss of the sample during plasma irradiation. The weight change of the sample was measured using an electric scale (model Pioneer PA214C) with a sensitivity limit of 10^{-4} g. After oxidizing the cobalt on the surface, the samples are washed in a bath of distilled water and acetone to remove any unoxidized cobalt. Subsequently, the samples are dried and reweighed using a scale. Each sample is then exposed to plasma radiation for 2 minutes in 4 steps. After irradiation, any Cobalt powder separated from the surface is cleaned using a blower, and the sample is reweighed using the same scale to determine the cleaning speed.

The surface of the samples was analyzed before and after the reaction using EDX (Energy Dispersive X-ray Spectroscopy), OM (Optical microscope), and SEM (Scanning Electron Microscope) analysis. The results of the EDX and SEM analysis after plasma irradiation are presented in Fig. 6. Additionally, Table 3 provides further details on these results. The OM images are also included to supplement these findings in Fig. 7.

Table 3: Results of EDX analysis after plasma irradiation on the sample surface.

Element	Weight (%)	Atomic (%)
O	4.02	12.66
Cr	18.85	18.26
Fe	65.75	59.31
Co	1.36	1.16
Ni	10.03	8.61

Table 4: Comparison of different methods of cobalt decontamination with plasma.

Discharge	Discharge conditions	Input power	Pressure	Plasma	Gas	Mat	Etching rate (μmol.min^{-1})	Ref.
RF	13.56 MHz	220 W	0.45 torr	Low Temp	NF_3	Co_3O_4	6.69	(Lee et al., 2019)
Micro wave	2.54 GHz	1.5 kW	1 atm	Thermal	CF_4-O_2	Co_2O_3	625	(Windarto et al., 2000b)
DBD	6.5 kV 3 kHz	7 W	1 atm	Low Temp	70% He 24% CF_4 6% O_2	Co_2O_3	10	(Suzuki et al., 2005)
DBD	4.5 kV 25 kHz	20 W	1 atm	Low Temp	95% He 5% F_2	Co_3O_4	10.875	Our study

3 Results and discussion

As stated in Eq. (1), it was expected that after plasma irradiation, the cobalt oxide layer sample would be pulverized and separated from the sample surface. This is exactly what happened and is visible by the naked eye in Fig. 8. The produced powder can be easily collected from the surface using a simple vacuum surface sweeper.

The samples were irradiated with plasma for 2 minutes and then weighed to calculate the Cobalt etching rate from the weight change, our results showed that after 6 minutes, 88.23% removal was achieved and its value remained almost constant, which is shown in the diagram of Fig. 9. The surface morphology changes shown by SEM, EDX, and optical microscopy analysis also confirm this etching velocity measurement, see Figs. 6, 8, and 9, respectively.

In order to check the properties of the removed powder, it was necessary to perform XPS and AES analysis, which unfortunately they were not available, but this gap has been tried to be compensated by performing EDS analysis on the surface of the sample before and after the test. As it is concluded from results presented in Tables 2 and 3 the Co Atomic% reduce from 22.21% to 1.16%.

Decontamination rate is defined as equal to:

$$\text{Rate } (\%) = \frac{x_0 - x_t}{x_0} \times 100, \tag{5}$$

where x_0 and x_y are quantities of cobalt present on specimen surface respectively before and after decontamination.

Also, the etching rate was calculated and its value was 10.875 ± 0.052 micromoles per minute for this generator. The location of the thermocouple is shown in Fig. 7 and its value was 300 Kelvin. This temperature does not cause any damage to the substrate metal and this can be an important factor in removing contamination from the surface without degrading the substrate metal.

Table 4 presents a comparison of this research findings with other researchers who have conducted similar work in the same etching rate unit. Jaeyong Lee (Lee et al., 2019) reported the etching rate in micrometers per minute, where they measured the thickness decrease. The properties of cobalt (II, III) oxides are presented in Table 2 of reference (Lee et al., 2019), with a molar mass of 240 g.mol^{-1} and a density of 6.11 g.cm^{-3}. The samples used were discs measuring 20 mm in diameter and 2 mm in thickness. Using this information, an etching rate of 3.36 μmol.min^{-1} corresponds to 6.69 μmol.min^{-1}. In contrast, Windarto (Windarto et al., 2000b) used samples with a surface area of 25 mm × 25 mm. Therefore, an etching rate of 100 μmol.cm^{-2}.min is equivalent to 625 μmol.min^{-1}.

In the end, it was concluded that this method can be used as a suitable alternative to mechanical and wet methods of cobalt decontamination of metal surfaces that have disadvantages such as degradation of the substrate metal or the production of large amounts of secondary liquid waste. It is also clear from Table 4 that the atmospheric pressure process is more suitable in terms of speed and cost due to the higher etching speed and no need for a vacuum chamber.

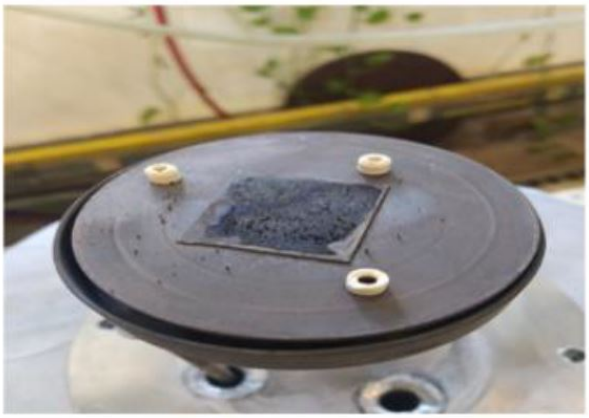

Figure 8: Cobalt oxide lifted from the sample surface after irradiation with DBD plasma resulting from a mixture of fluorine and helium gas with an input current of 300 SCCM, at a voltage of 4.5 kV and a frequency of 25 kHz.

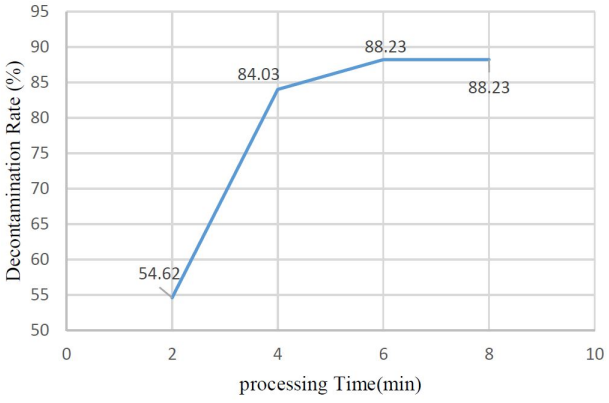

Figure 9: Dependence of decontamination ratio on process time.

Also, from the economic point of view, the process of atmospheric pressure surpasses the process of low pressure. The first rate is one or two times higher than the second. Therefore, the former is superior for cleaning large areas such as the inner surfaces of reactor vessels. Also, it is easy to install DBDs in the pipes of a steam generator. The ratio of H rate to input power can be used as one of the economic indicators. Although the microwave process has a good etching speed, it consumes a lot of energy. The DBD process requires only a small amount of power and can undoubtedly be ranked high, although additional heating is required for the process. Other processes require additional energy to remove fluoride. It can be concluded that the non-equilibrium plasma process has a special feature that can provide a large number of active atoms for the selective reaction without generating additional heat.

4 Conclusions

The OECD/NEA has recently claimed that approximately 30 million tons of contaminated scrap metal will be generated over the next 50 years from the decomposition and decommissioning of nuclear facilities (Chen, 1997). The International Atomic Energy Agency predicts that 650 tons of radioactive metal waste will be generated due to the deactivation of PWR with a capacity of 900 to 1300 MW (Coates et al., 2008). Most of the metallic wastes are surfacecontaminated. Therefore, developments of efficient surface decontamination techniques are international concern to minimize the waste generation. Co-58 and Co-60 are the most troublesome radioactive nucleotides that are cleared in many cases, which usually exist inside the oxide film on the base metal surface.

Techniques for cleaning or etching metal surfaces using reactive plasma gas are emerging as one of the dry processing techniques which are for surface contaminants with high bond energy, especially used to clean used parts and equipment (Kim et al., 1999; Nam and Um, 2022; Kim et al., 2004, 2003a,b; Windarto et al., 2000a; Mehrabifard, 2023; Omrani et al., 2023). This method can maintain the need for minimal secondary waste production while maintaining the same efficiency level of wet cleaning techniques (Kim et al., 2004; IAEA, 1999). In principle, this method selectively selects surface contaminants, converts them to volatile compounds through the catalytic surface, and finally removes them from the surface (Kim et al., 1999; Nam and Um, 2022; Kim et al., 2003a,b; Windarto et al., 2000a; Mehrabifard, 2023; Amakawa, 2001; Toumanov, 2003; Girold and Barthelemy, 2001; Martz, 1991).

In this study, the plasma reaction due to the discharge of a dielectric barrier of a mixture of 95% helium and 5% fluorine with cobalt oxide film (Co_3O_4) grown on the surface of stainless steel 304 was studied experimentally. Experimental results show that cobalt oxide becomes a powder after plasma irradiation and is easily separated from the surface of the base metal. The optimal plasma generating conditions of the dielectric barrier discharge used in this experimental study were obtained at atmospheric pressure, voltage 4.5 kV, and frequency 25 kHz with a etching rate of 10.875 μmol.min^{-1}. The samples were analyzed before and after plasma irradiation, using SEM/EDX (Scanning Electron Microscopy with Energy Dispersive X-ray Xpectroscopy) and the purification rate was performed using a sequential weighting of the samples with scales 10^{-4} g accurately obtained.

In conclusion, the results of this study demonstrate that the plasma decontamination technique can be applied to efficiently and effectively remove major radioactive surface Contaminants of cobalt, hiding in the oxide on the surface of metallic stainless steel 304 generated during decommissioning of old nuclear power plants.

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Conflict of Interest

The authors declare no potential conflict of interest regarding the publication of this work.

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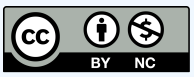

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