Modern renium reduction methods and hydrometallurgical approaches and advanced plasma technologies

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Abstract. The article provides a detailed analysis of modern methods for reducing rhenium, including traditional hydrometallurgical approaches and advanced plasma technologies. The thermodynamic and kinetic aspects of reduction processes, which determine the efficiency of obtaining highly pure rhenium, were considered. Experimental data confirming the plasma effect on the morphological and structural characteristics of the obtained metallic rhenium are presented. Particular attention is paid to the analysis of impurity composition, process energy parameters, and the prospects for the industrial implementation of plasma technologies in the metallurgy of rare metals.

INTRODUCTION

Reduction of ammonium perrenate with hydrogen using plasma. Since there is no data in open sources on the hydrogen reduction of rhenium from ammonium perrhenate using plasma, let's consider the closest processes - the reduction of metals from oxides. Most metals, such as iron, aluminum, silicon, manganese, chromium, nickel, titanium, vanadium, etc., are extracted from oxide minerals by reduction.

Charged particles in plasma play a significant role. Reduction processes occur at the plasma-substrate (tablet) powder interface where the reducing gas is in a plasma state. [2] Plasma, which conducts electricity, is in contact with the processed material, which is in a solid or liquid state. Obviously, then the polarity of the charge must play an important role in attracting the necessary particles from the plasma volume to the interface where the desired reactions occur.For example, for reduction from iron oxide, the positive polarity of the reacting surface reduces the thermodynamic potential of the reduction reaction compared to the case with a neutral reacting surface. Conversely, in the case of a surface with negative polarity, the reaction proceeds much faster, i.e., ΔG is more negative [8
For a chemical reaction to occur, the reacting particles with sufficient kinetic energy must first collide to overcome the activation barrier. In the context of plasma interaction with metal oxides, the role of vibration-excited hydrogen molecules is emphasized in the literature. Molecules in a state of vibrational vibration can have internal energy up to 4.5 eV, which is obtained during rotation and vibration. They transfer their internal energy to other molecules and atoms in the gas phase during inelastic collisions and chemical reactions. As a result, the internal energy of the reacting particles increases, and the activation barrier decreases, facilitating the reaction. [12]

Hydrogen plasma not only provides improved reduction conditions but also provides high plasma temperatures for intensifying reduction processes, thereby enabling processes to be carried out in a single stage, which, in turn, makes metallurgical equipment smaller in size compared to traditional equipment.

EXPERIMENTAL RESEARCH

When reducing ammonium or potassium perrenates with hydrogen, rhenium powder is obtained. Electrolytic isolation yields rhenium powder or flakes (spongy rhenium precipitates). Rhenium coatings are produced through thermal dissociation. [1]

Reduction of potassium and ammonium perrenate with hydrogen under pressure (in solutions and solid state). It has been established that potassium and ammonium perrenates, both in solution and in solid form, are reduced by hydrogen at relatively low temperatures (200-300°C) under 20-100 atm pressure. [11] When water-free potassium or ammonium perrenates are reduced with hydrogen under a pressure of 50 atm, metallic rhenium is obtained in just 1-2 hours. [6]

When reduced by hydrogen under the pressure of potassium or ammonium perrenate solutions in the presence of hydrochloric acid, a number of low-valence intermediate rhenium compounds can be obtained, for example, (NH4) 2ReCl6 and (NH4) 2ReCl4, which can be used to obtain high-purity, oxygen-free metallic rhenium. [9] Reduction proceeds according to the following reactions:

$$2NH_4ReO_4 + 3H_2 + 12\ HCl = (NH_4)_2ReCl_6 + H_2ReCl_6 + 8H_2O \text{ (at 270°C);} \qquad (1)$$

$$H_2ReCl_6 + 2NH_4Cl = (NH_4)_2ReCl_6 + 2HCl; \qquad (2)$$

$$(NH_4)_2ReCl_6 + H_2 = (NH_4)_2ReCl_4 + 2HCl \text{ (at 300 – 325°C in solution HCl); [4]}$$

The creation of an experimental installation designed to carry out reduction processes for obtaining high-purity rhenium on hydrogen plasma was preceded by the study of the results of our own experimental studies, which allowed us to form a list of the main technical requirements for the installation. Physical processes in plasma strongly depend on the geometric parameters of the reactor, such as the distance between the electrodes and the size of the electrodes, which has a huge influence on the field strengths between the electrodes. In general, the plasma-chemical reactor was a quartz tube placed in a muffle furnace with open ends and an electrode system (Fig.2).

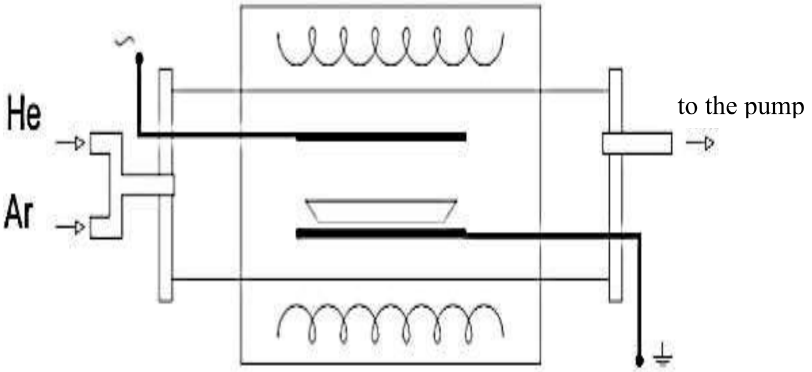

FIGURE 1. Principal diagram of a plasma chemical reactor

The reactor's vacuum system is equipped with a mechanical pump and a cooling trap. The specific vacuum obtained in the chamber is 10-2 torr. Vacuum is measured by vacuum gauges in the chamber and the vacuum line. The gas system has two nodes for supplying hydrogen and inert gas.

The samples were studied more thoroughly using Raman spectroscopy. Below, Figures 3-6 show the Raman spectra of ammonium perrenate (for comparison) and samples that show the disappearance of characteristic peaks of ammonium perrenate, confirming the complete reduction of metallic rhenium from ammonium perrenate [3-40]. Some "noise" peaks in the spectra are characterized by the post-process oxidation of the surfaces of fine rhenium metal powders. The absence of such noise in the X-ray diffraction spectra confirms this fact.

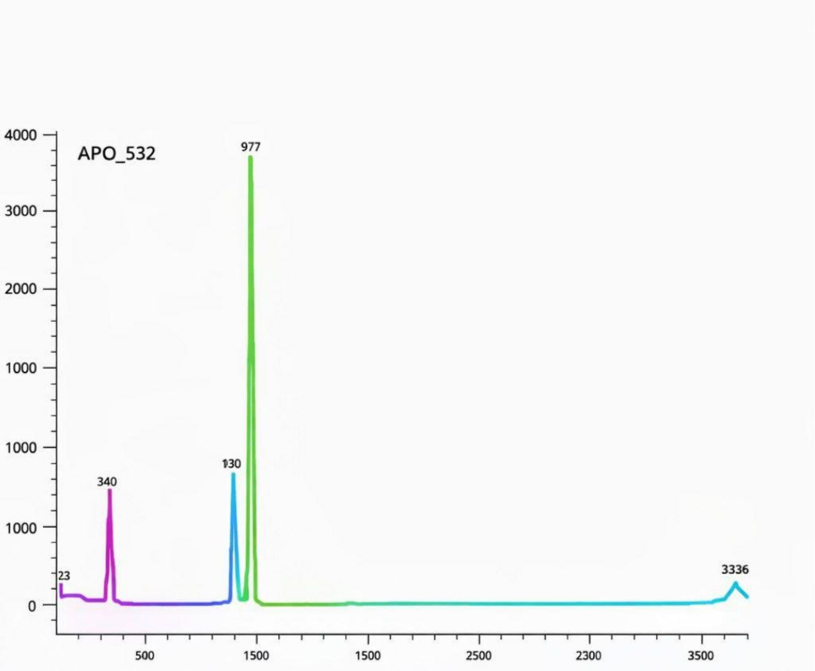

FIGURE 2. Ammonium perrenate Raman spectrum

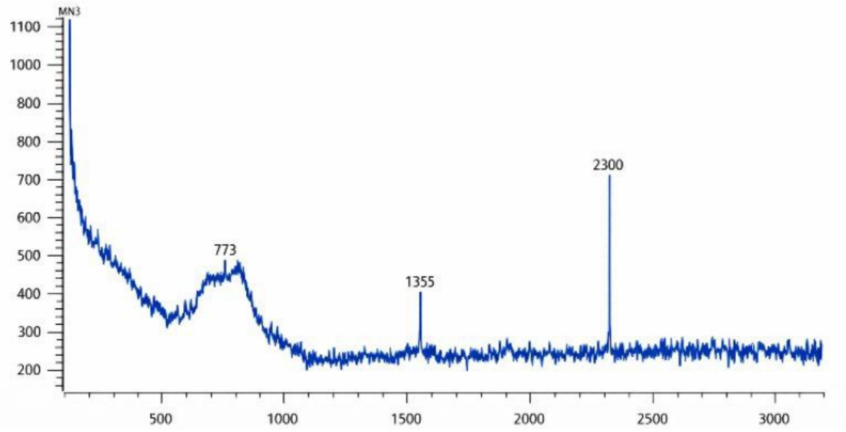

FIGURE 3. Rhenium metal sample Raman spectrum

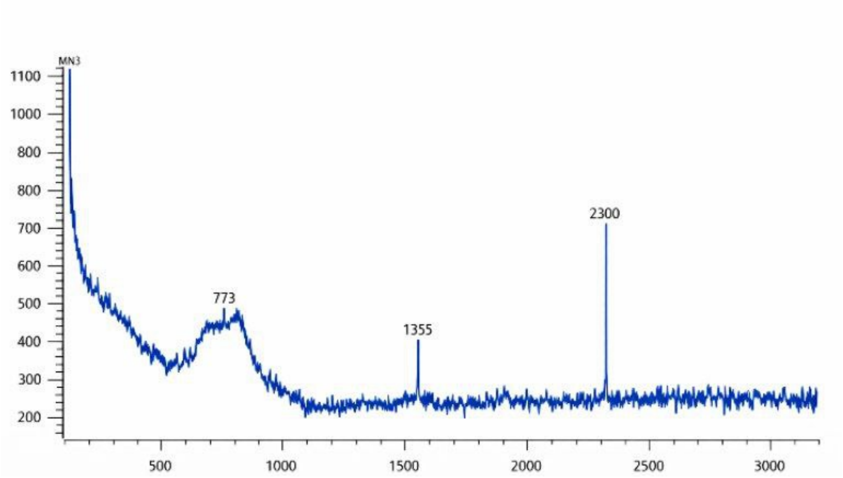

FIGURE 4. Rhenium metal sample Raman spectrum

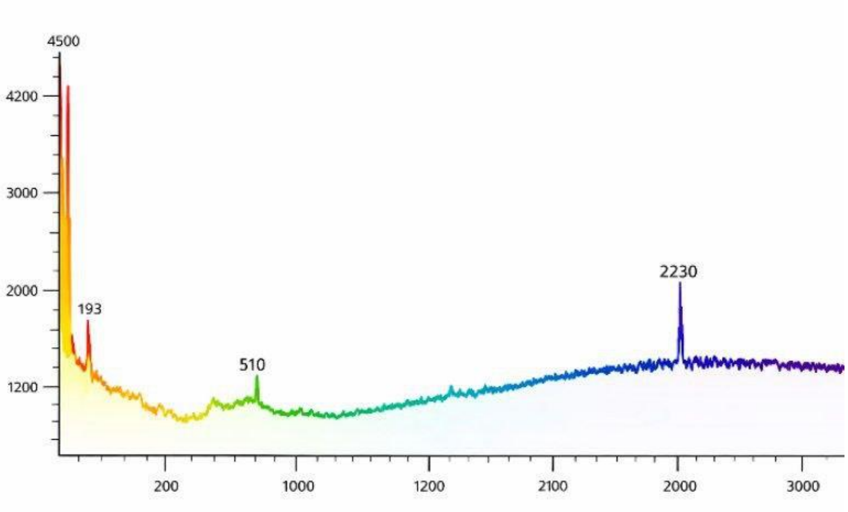

FIGURE 5. Rhenium metal sample Raman spectrum

RESEARCH RESULTS

The "chlorine" method also includes the method of chlorinating technical rhenium at a temperature of 600-700°C. The resulting rhenium pentachloride, ReCl5, is purified by vacuum distillation. Chloride is decomposed with water. In this case, 65-70% of rhenium precipitates as hydrated dioxide. Complete decomposition of chloride is achieved at pH=2.5-3.0. The filtered and washed precipitate is dried in a vacuum and reduced with hydrogen in two stages: first at 400-600°C, then at 800°C. The solution is oxidized with hydrogen peroxide, neutralized with ammonia, and ammonium perrhenate is extracted from it, which is returned for reduction. The method provides good cleaning from impurities of alkaline and alkaline-earth metals. At the same time, iron, silicon, and a number of other impurities are not separated.

Data on the content of impurities in the powder of rhenium obtained by various methods of reduction are presented in Table 1.

TABLE 1. Content of impurities in rhenium powder obtained by various methods

Impurities	Content of impurities in the obtained rhenium (%)		
	from $KReO_4$	from NH_4ReO_4	chloride method
Silicon	0,005	0,028	0,015
Iron	0,050	0,024	0,012
Aluminum	0,009	0,094	0,008
Manganese	0,007	0,002	0.007
Magnesium	0,002	0,038	0.005
Calcium	0,008	0,017	0,002
Copper	0,001	0,002	0,0002
Molybdenum	0,150	No	No
Potassium	0,410	No	No
Sodium	0,150	No	No
Nickel	0,003	No	No
Chromium	0,004	No	No
Total impurities:	0,799	0,205	0,049

Thus, the obtained results show that plasma contributes to a more efficient process of reducing rhenium from ammonium perrenate. This leads to a noticeable increase in process productivity, a decrease in energy consumption, and a reduction in hydrogen consumption. A detailed analysis of the thermal and plasma reduction processes for rhenium shows that the use of plasma allows for a two-fold reduction in the cost per unit of electrical energy product and a three-fold reduction in the cost per unit of hydrogen product. The total productivity of the units can also be increased by 60- 100% by increasing the thickness of the ammonium perrhenate loading layer. At the same time, no noticeable changes in the granulometric composition of the rhenium metal are observed; apparently, the granulometric composition is largely determined by the characteristics of the initial ammonium perrenate powder.[5]

Regarding the samples in Figures 4-6 reduced by superpure hydrogen in the presence of plasma, impurities such as Rb - 168.2 ppm, Sb - 54.8 ppm, P - 23.3 ppm, Te - 9.5 ppm, Au - 18.1 ppm, Pt - 13.7 ppm, S - 6.4 ppm are noticeable. [10] The rhenium content in these samples is determined from 99.94% to 99.96%, which is also closer to the Re-1 grade. However, relatively high sulfur content from 0.0003 to 0.005% is found in the samples. This circumstance requires the development of additional measures to remove sulfur from the product composition.

CONCLUSIONS

Analysis of the presented methods for reducing rhenium shows that the use of plasma technology allows for a higher degree of metal extraction (95-98%) while reducing energy costs and minimizing losses. Compared to traditional methods, plasma reduction contributes to the improvement of the physicochemical characteristics of rhenium, including its purity and granulometric composition. Further development of this technology requires in-depth study of the mechanisms of rhenium-plasma interaction, improvement of impurity removal processes, and optimization of conditions for large-scale industrial applications.

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