Separation of Niobium From Nickel-Chromium-Iron Containing Nickel-Based Alloys

Kyungwon Suh* and Youngkyong Ha
Nuclear Chemistry Research Division, KAERI, 111, Daedeok-daero 989beon-gil, Yuseong-gu, Daejeon, Republic of Korea
*kyungwon0707@kaeri.re.kr

1. Introduction

Nickel-based alloys have been extensively used and been one of the important materials in nuclear facilities because of their high resistant to aqueous corrosion in a variety of environment and their resistant to high temperature. These alloys can be applied to a nuclear structure tube such as steam generator tube for PWRs and for some CANDU reactor. They can also be used as fuel element cladding and subassembly wrappers due to the high resistance to radiation-induced void swelling. Recently, there is a revival of interest in these alloys for Generation IV reactor and molten salt reactor, as well as in consideration of their use for the pressure vessel of the Prometheus space reactor [1].

Niobium is one of the alloying elements in nickel-based alloy. With the presence of niobium, it improve the intensity at high temperature, the temper brittleness and sensitivity to superheat even though less than 0.1% of niobium. In other words, the addition of niobium to nickel-based alloy provides solid solution strengthening, strengthening through carbide formation and precipitation hardening as well [2].

In addition, niobium is present as impurity in structural component of nuclear reactor pressure vessel and the cladding of nuclear fuel. By neutron activation of the stable niobium, the long-lived radioactive niobium-94 was generated. The radioactive concentration of niobium-94 was too low to direct gamma-measurement. Therefore, it is important to separate niobium from other elements and to quantity the amount of niobium present in nickel-based alloy for modification of thermal properties in nickel-based ally.

In this paper, the procedure for separation of niobium from nickel, chromium, and iron containing nickel-based alloy was demonstrated using following method: precipitation, solvent extraction, and anion exchange chromatography.

2. Experimental

2.1 Reagents

All reagents were commercially available and used without further purification. The niobium standard solution was from Accustandard. The nickel, chromium, and iron solution were prepared with following reagent.

Table 1. Each element composition

Element	composition
Ni	Ni powder and HNO_3
Cr	$Cr(NO_3)_3$ and HNO_3
Fe	$Fe(NO_3)_3$ and HNO_3

MIBK (methyl isobutyl ketone) was purchased from SHOWA. Anionic exchange resin (AG 1-X8) was from BioRad.

2.2 Method

Each metal solution was mixed to prepare the simulation sample, which contained 150 mg nickel, 30 mg chromium, 20 mg iron, and 0.2 mg niobium.

For a niobium separation, main elements like nickel, chromium, and iron should be selectively isolated. First, precipitation was applied to lower the nickel content by adding 10% NH_4OH.

Second, iron was isolated by MIBK extraction from the precipitate containing nickel, chromium, iron, and niobium generated at precipitation step.

Last, niobium was separated from chromium and nickel using anion exchange chromatography.

Each element was determined by measurement of ICP-AES (Horiba Jobin Yvon, Activa M) in each step.

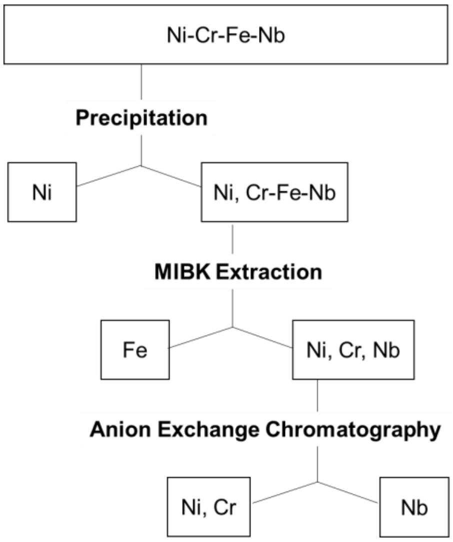

Fig. 1. Scheme for Separation.

3. Results

3.1 The precipitation with 10% NH_4OH

A different volume of 10% NH_4OH was added to the simulated solution for an isolation of the nickel. Although chromium, iron and niobium were precipitated regardless of the volume of added NH_4OH, the nickel content was dependent on the volume of NH_4OH. In Fig. 2, the nickel content was the highest in a solution at 0.8 mL of 10% NH_4OH, which meant that was the lowest in a precipitate.

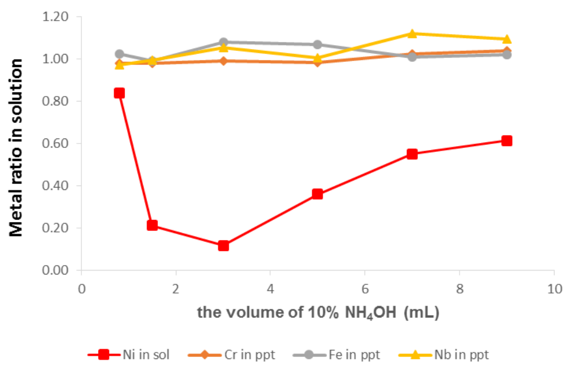

Fig. 2. Metal contents in precipitation step.

3.2 Solvent extraction with MIBK

There were nickel, chromium, iron, and niobium in a precipitate after the precipitation. Iron was extracted at the combination of MIBK and HCl [3]. For solvent extraction, the precipitate was dissolved in 4 M HCl and dissolved solution was mixed with MIBK. Iron was isolated from nickel and chromium, however, niobium recovery was 60 ~ 70%.

3.3 Anion exchange chromatography

Anion exchange chromatography was applied to separate niobium from nickel and chromium after MIBK extraction. A solution containing nickel, chromium, and niobium was loaded to anion resin (AG 1-X8) pretreated with 8 M HCl, the column was washed with 8 M HCl, and niobium was stripped 14% NH_4Cl and 4% HF mixture.

4. Conclusion

In here, the procedure for separation of niobium from nickel-based alloy containing nickel, chromium, and iron was demonstrated. First, precipitation was applied to lower the nickel content following MIBK extraction to isolate iron. Last, anion exchange chromatography was utilized to separate niobium from nickel and chromium. Although the final recovery of niobium was moderate, niobium was separated from nickel, chromium, and iron. This procedure can be applied to separate niobium in a radioactive metallic waste.

REFERENCES

[1] “Comprehensive Nuclear Materails”, 1st Ed, R. Kongins, R. Stoller, T. Allen, and S. Yamanaka, Eds., Elsevier, 2012.

[2] G. D. Smith and S. J. Patel, “The role of niobium in wrought precipitation-hardened nickel-base alloys”, International Symposium on Superalloys 718, 625, 706, and Derivatives, TMS, 2005.

[3] J. Pinajian, “The methyl isobutyl ketone extraction of trivalent iron from hydrochloric acid solution and the effect of an inert dilution on the distribution coefficient” (1966).