

High-temperature metal corrosion tests for HI decomposer

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1. Introduction

The Sulfur-Iodine thermochemical Nuclear hydrogen production process is composed of three parts, Bunsen reaction, sulfuric acid decomposition reaction and hydriodic acid decomposition reaction. Among them, hydriodic acid decomposition reaction has low kinetics and equilibrium yield is poor, being an efficiency-determining step.¹⁾ Thus, many efforts are tried to raise the reaction rate and yield, such as extractive/reactive distillation or EED method. High temperature decomposition process,²⁾ another candidate of HI decomposition method nowadays, has a simple process but due to highly corrosive environment, a material problem is one of crucial obstacles. In this paper, a number of structure material candidates are tested at high temperature for HI decomposition process.

2. Methods and Results

In this section, material selection strategy and test results are described. Test results include the mass reduction rate, XRD and SEM analysis.

2.1 Material selection

For the test material selection, we did a screen process for lots of alloys following some criteria. First, the noble metal should be excluded due to its high cost. Second, commercially produced metal is proper to be tested and practically recommended.

There are SI process structure material candidates already suggested by the former researchers, including the HI decomposition process at the 500°C. S. Kasahara et al. suggested that MAT21 and Hastelloy are metals for the HI decomposition process and Incoloy800 and Hastelloy for the sulfuric acid decomposition.³⁾ Referring to them, we selected Incoloy800HT and HastelloyC276, because the sulfuric acid decomposition reaction is high temperature process. Additionally, SS446 is considered as a good candidate due to its corrosion and heat resistivity.

2.2 Corrosion test procedure

The metal corrosion test is performed at 850°C in gas phase HI flow. An experimental apparatus is shown in Fig.1. Hydriodic acid is fed to the evaporator by a micro pump and mixed with carrier gas and moves to the quartz reactor enclosed by the electric furnace. Metal

coin samples are placed at the center of quartz reactor supported by quartz wool.

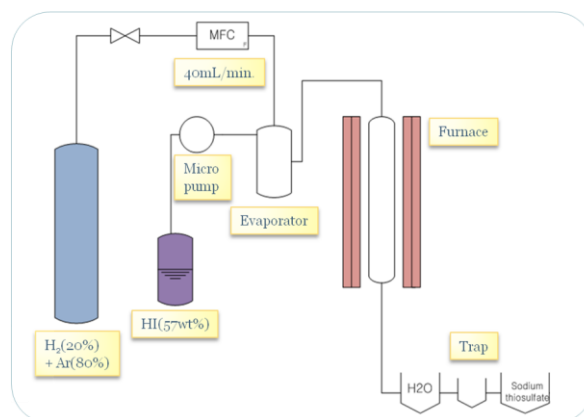


Fig. 1. Experimental apparatus for corrosion test

Corrosion tests are performed twice. The first test was done for 85hrs with 0.23ml/min of the HI flow rate. During the test, samples are weighed for 5 times. The second test was done for 307hrs with 0.21ml/min of the HI flow rate. In this experiment, samples are weighed only 1 time after whole the test is finished.

2.3 Test results and analysis

Mass reduction rate for the first test is shown in Fig. 2.

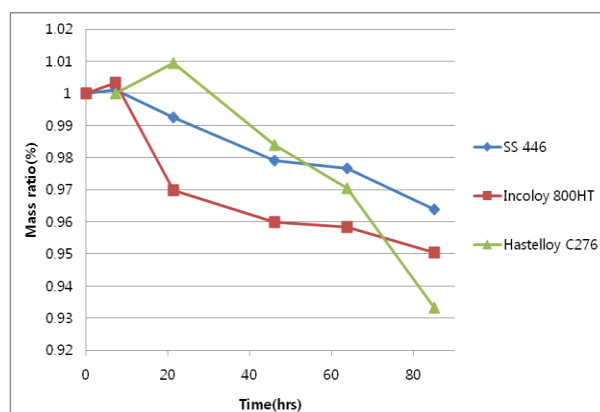


Fig. 2. Mass reduction rate of first corrosion test

As shown in Fig.2, SS446 and Incoloy800HT show 3-5% mass reduction and HastelloyC276 lost 7% of total mass after 85 hrs test. Second test result shows the mass reduction rate of 9.3% for the SS446, 8.1% for the Incoloy800HT and 38.2% for the HastelloyC276. From these results, HastelloyC276 would be excluded from structure material candidates by its high mass reduction

rate. Incoloy800HT and SS446 have similar mass reduction rates after the longer term test, so surface element and cross section analysis were additionally performed.

To analyze the surface element change, XRD analyses on tested samples are conducted. XRD peaks of the HastelloyC276 show chromite($\text{Fe}^{+2}\text{Cr}_2\text{O}_4$) and eskolaite which mean that thick outer oxide layer exists. XRD peaks of the SS446 and the Incoloy800HT show not only the peaks of the Hastelloy's XRD result but also manganese from the SS446 and Iron Nickel and so on from the Incoloy800HT. So it can be assumed that the oxide layers of SS446 and Incoloy800HT are thinner than that of HastelloyC276, which is mostly unstable.

Lastly, SEM analysis on the cross-section of the secondly tested samples is conducted. Fig. 3 shows a cross section of the HastelloyC276. It shows mostly broken oxidation layer and deep-cross linking internal oxidation. It can be said that this metal lost big part of its base metal and the internal oxidation is severe with HI gas in high temperature.

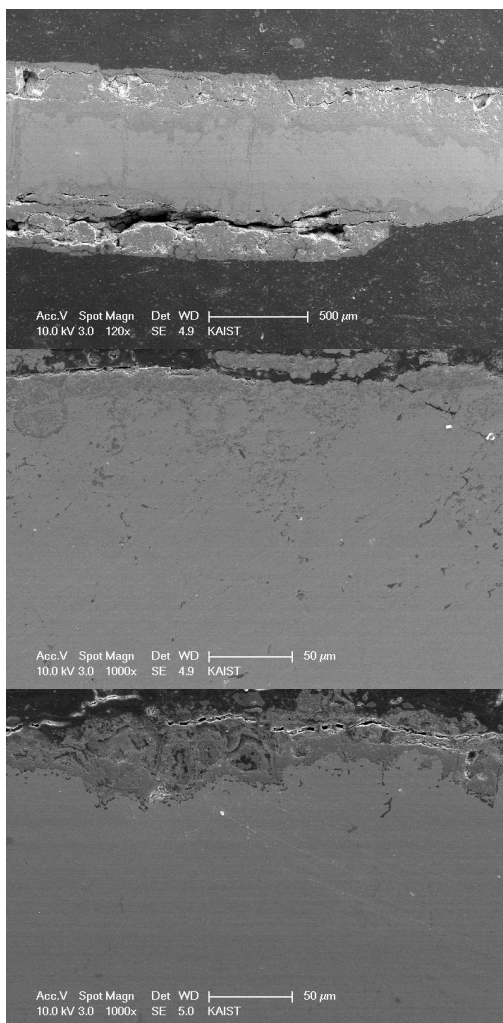


Fig. 3-5. SEM image of cross section of HastelloyC276(top), Incoloy(middle) and SS446(bottom) after test

From Fig. 4, by EDAX analysis, dots inside the base metal are silicon/aluminum oxide-rich, which are internal oxidation causing elements. Thus, it is shown that the Incoloy800HT has serious internal oxidation region. This situation is guessed to be caused by the nickel ingredient, because nickel content was the highest when we analyzed leftover at the outlet of the quartz reactor. Surface oxidation layer was also easily detached. From Fig. 5, SS446 showed disorder and mostly detached surface oxidation layer, too. Beyond the surface oxidation layer, there were internal oxidation spots, but not those much like the Incoloy800HT or HastelloyC276. This difference is, as stated, supposed to be due to an absence of the nickel in SS446.

3. Conclusions

We tested three commercial metal samples to find a proper candidate for the high temperature HI decomposition process, but all samples showed high mass reduction rate and unstable surface oxidation layer. There were obvious differences among three metal samples. The HastelloyC276 showed extremely poor characteristics and the Incoloy800HT had lots of internal oxidation spots. The SS446 has the discontinuous and unstable surface oxidation layer, but its internal part was relatively stable.

Therefore, we can expect that it can be a good practical candidate for the high temperature HI decomposition process if the proper protective layer can be added or surface modification is possible on the SS446.

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