Development of Manufacturing Technology for High Purity Low Activation Vanadium Alloys

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Abstract. Vanadium alloys are promising candidate low activation materials for structural components of fusion reactors. Establishment of industrial infrastructure is, however, remaining to be a critical issue because of lack of other large scale commercial applications. In the present study, technologies for large scale manufacturing of high purity V-4Cr-4Ti alloy were developed by improving the present commercial production processes of vanadium metal, and optimizing alloying, plating, sheeting and wiring techniques. Efforts were focused on reducing carbon, nitrogen and oxygen impurities, which are known to deteriorate workability, weldability and radiation resistance of vanadium alloys. Especially, improvements were made in atmospheric control during calcination, aluminothermic reduction, vacuum arc remelting, and hot forging and rolling. A medium size (30kg) high purity V-4Cr-4Ti ingot was produced and designated as NIFS-HEAT-1. The specimens produced out of the ingot are being submitted to Round-robin tests by Japanese universities. Two larger ingots of 166kg in total weight were produced recently (NIFS-HEAT-2(A) and (B)). By these efforts, technology for fabricating large V-4Cr-4Ti alloy products with <100ppm C, ~100ppm N and 100~200ppm O was demonstrated.

1. Introduction

Vanadium alloys are promising candidate fusion structural materials because of their low activation properties, high temperature thermomechanical properties, good resistance against neutron irradiation and excellent compatibility with liquid Li[1,2]. However, establishment of industrial infrastructure is remaining to be a critical issue for vanadium alloys, because of lack of other large scale commercial application.

Recent studies on vanadium alloys showed that the increase in interstitial impurities such as carbon, nitrogen and oxygen results in loss of workability[3], degradation of weldability[4] and enhanced loss of elongation by irradiation[5]. Thus, in the development of technology for large-scale production of vanadium alloys, suppression of the impurity levels is essential. US-DOE had a program for casting 500 and 1200 kg ingots of V-4Cr-4Ti alloy for fusion research [6,7]. The products fabricated from the ingots were characterized with and without irradiation. Those ingots had impurity levels of <100ppm C, 50-150ppm N and 300-400ppm O.

National Institute for Fusion Science (NIFS) is promoting a program for large scale manufacturing of a V-4Cr-4Ti alloy by collaboration with Japanese industry[8]. The program aims to demonstrate technical feasibility of fabricating a large ingot of high-purity V-4Cr-4Ti alloy. Also intended are to investigate impurity transportation during the fabrication process and to utilize the resulting ingots for Round-robin tests by Japanese Universities. This paper is an overview of the progress in the program.

2. Development of Manufacturing Technology
2.1 Purification of the Present Commercial Vanadium Metal

In this program, carbon and nitrogen levels of the present commercial vanadium metal were reduced by improving the manufacturing processes, first of all. Fig. 1 compares the conventional and the improved process of the vanadium metal production. Particular efforts were made for inhibiting nitrogen pick-up during the calcination and the aluminothermic reduction processes. Also improved was the purity of the Al reduction agent. By these improvements, carbon and nitrogen levels were reduced from ~300 to ~100ppm and from 300-700ppm to ~100ppm, respectively.

2.2 Alloying into V-4Cr-4Ti

Several alloying techniques were investigated to produce V-4Cr-4Ti minimizing the impurity pick-up. The Vacuum Arc Remelting (VAR) technique with some improvements in atmosphere was selected as the most suitable method of alloying. A 30kg V-4Cr-4Ti ingot was made by the VAR technique, whose impurity levels were ~60ppm C, ~100ppm N and ~180ppm O. The alloy ingot was designated as NIFS-HEAT-1.

2.3 Fabrication into Plates, Sheets and Wires

The ingot was canned into a case of 304 stainless steel followed by hot forging, hot rolling and cold rolling. The geometry of the case, especially the thickness of the edge part, was carefully designed so as not to break during the deformation. Plates of 6.6 and 4.0 mm thick and sheets of 0.25 mm thick were made out of the ingot. Also fabricated were wires of 2.0mm diameter for welding tests. Some of the plates and wires produced are shown in Fig.2.

The thermal treatment condition was investigated to obtain homogeneous distribution of grains whose sizes range from 0.02 to 0.03 mm. 1273 K and 2 hours was selected as the optimized condition for the plates of 6.6 and 4.0 mm thick. The increase in the impurity level was small during the fabrication and the thermal treatment.

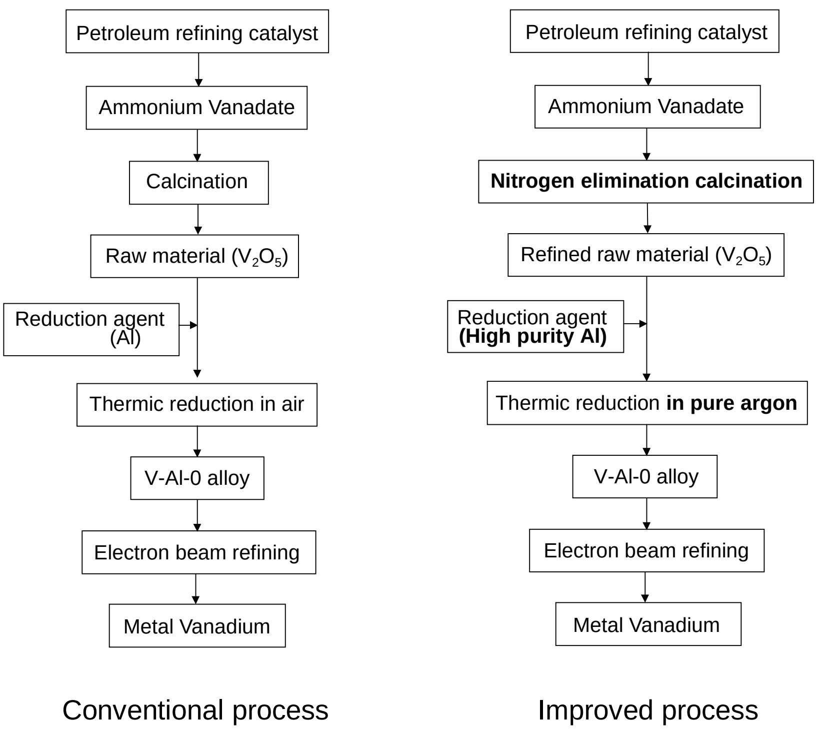


FIG.1. Conventional and improved production processes of vanadium metal.

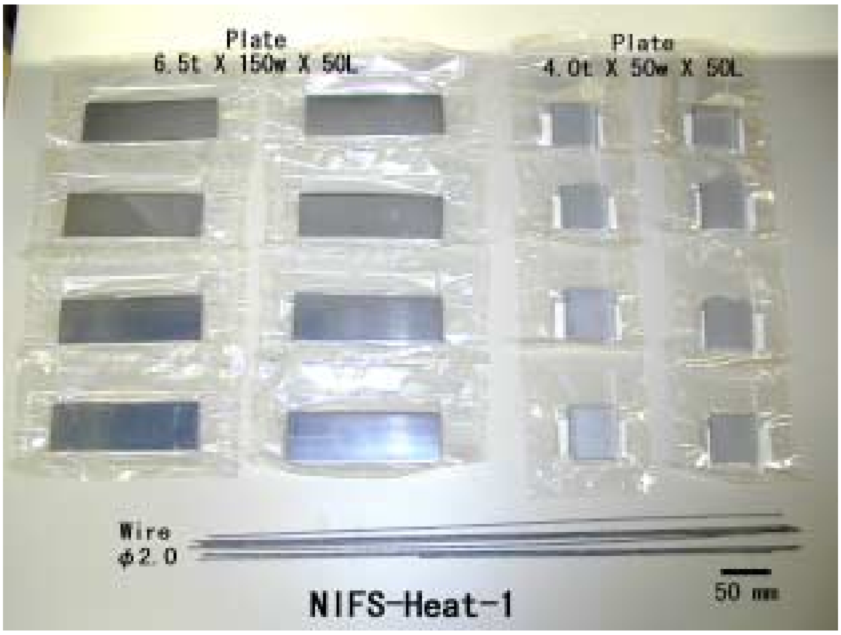


FIG.2. Plates and wires produced out of the high purity V-4Cr-4Ti ingot (NIFS-HEAT-1).

The nitrogen and oxygen contents of vanadium metal, V-4Cr-4Ti ingot and plates fabricated from the ingot are plotted in Fig. 3 for the present and the US-DOE fabrication(1200kg). The nitrogen and oxygen levels of the products of the present study are comparable to and almost half of those of the US-DOE program, respectively. In the US-DOE program, the rolling into plates and sheets was carried out at around 673K [7]. The fact that rolling of hot-rolled blocks to plates and sheets was possible at room temperature for the present alloy demonstrates a merit of simplifying the fabrication process by reducing the levels of interstitial impurities.

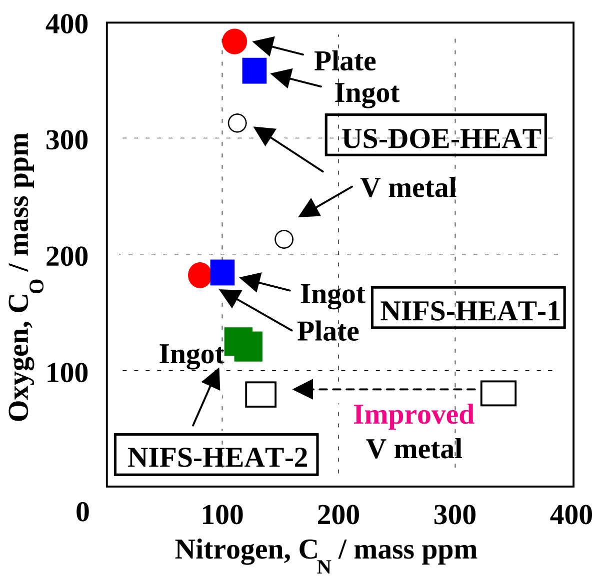


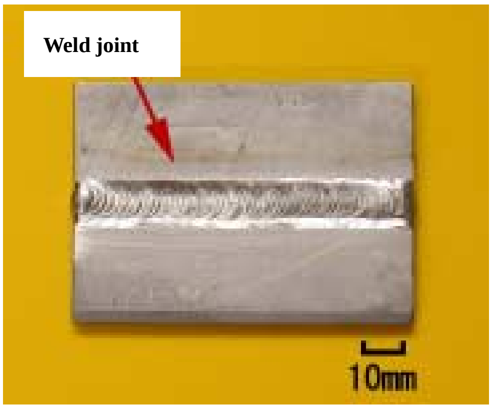
FIG.3. Impurity levels of vanadium metal, V-4Cr-4Ti alloy ingot (NIFS-HEAT-1 and NIFS-HEAT-2) and plates fabricated from NIFS-HEAT-1. The impurity levels of the products in the US-DOE program(1200kg ingot)[7] are shown for comparison.

Other minor impurities were detected by glow discharge mass spectrometry. The levels of Nb and Mo, which are particularly important elements because they produce long-lived radioactivity by fusion neutron irradiation, were estimated to be 6-7 and 20-25ppm, respectively. The Nb level of 6-7ppm is in contrast to 106ppm for the 1200kg ingot produce by the US-DOE program[7]. The high level of Nb in the US-DOE ingots was due to the fact that the facility used was also used for fabrication of Nb alloys, which is not the case for the present program.

The plates, sheets and wires fabricated from NIFS-HEAT-1 were submitted to the Round-robin tests by Japanese universities. The tests include chemical analyses, physical and thermal properties, mechanical properties, hydrogen retention properties, compatibility, performance as a plasma-facing material, key technology for component fabrication such as welding, coating, piping and so on. The products are also used for international collaborations. Welding tests were carried out, under the framework of Japan-USA collaboration program (JUPITER), at ORNL and ANL using Gas Tungsten Arc (GTA) welding and Laser welding techniques, respectively. Fig. 4 demonstrates the improvement in ductility of weld metals by reducing the oxygen level[9].

3. Fabrication of Larger Ingots

A second fabrication of ingots of 166kg in total weight was carried out by using the technology developed for the fabrication of NIFS-HEAT-1. They are a pair of ingots of 80kg and 86kg, designated as NIFS-HEAT-2(A) and (B), respectively. The outward appearance of the ingots after removing the surface layer is shown in Fig. 5. The impurity levels are O:106ppm and N:131ppm for NIFS-HEAT-2(A) and O:127ppm and N:126ppm for NIFS-HEAT-2(B). Thus the oxygen level of NIFS-HEAT-2 is even lower than that of NIFS-HEAT-1. The data are also shown in Fig. 3.



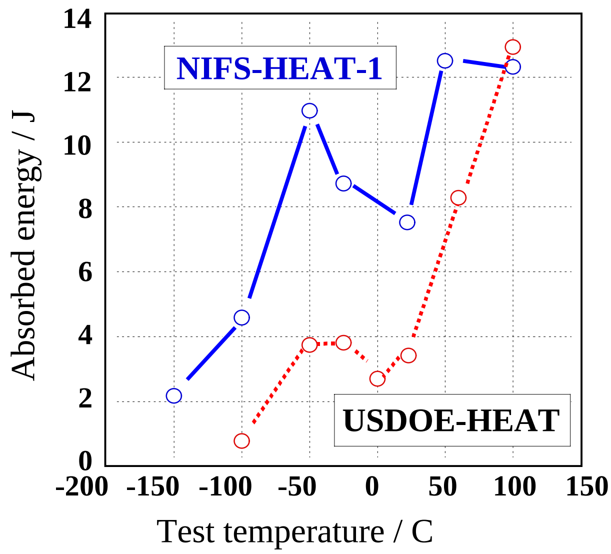


FIG.4. The GTA weld joint of NIFS-HEAT-1 (left) and the comparison of absorbed energy of the weld metal by Charpy impact tests for NIFS-HEAT-1 and USDOE-HEAT(500kg ingot[6])(right)[9].

FIG.5. V-4Cr-4Ti ingots produced by the second fabrication after removing surfaces. NIFS-HEAT-2(A) (top) and NIFS-HEAT-2(B) (bottom).

4. Conclusion

The technology for fabricating large V-4Cr-4Ti alloy products with <100ppm C, ~100ppm N and 100~200ppm O was demonstrated.

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