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Development of an ultra-fine grained V–1.7 mass% Y alloy dispersed with yttrium compounds having superior ductility and high strength

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Abstract

In order to develop an ultra-fine grained vanadium alloy dispersed with nano-sized dispersoids having superior ductility and high strength, powder metallurgical methods utilizing mechanical alloying (MA) and hot isostatic pressing (HIP) processes were applied to vanadium with 1.7 mass% yttrium addition. The developed alloy was annealed at 1273–1573 K for 3.6 ks and subjected to TEM observations and tensile tests at room temperature.

The alloy has average grain sizes from 330 to 750 nm, and average particle sizes of Y_2O_3 and YN from 7 to 23 nm, depending on annealing temperature. It exhibits superior ductility and high strength even in the as-HIPed state (no plastic working). Eighty percent cold rolling slightly improves the tensile properties. These results indicate that the microstructure of the alloy is almost free from weak portions associated with gaseous interstitial impurities that cause significant loss of ductility.

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1. Introduction

It is well known that vanadium is very chemically reactive with gaseous interstitial impurities, particularly nitrogen and oxygen. Dissolution of the interstitials into the matrix leads to significant hardening and ductility loss. In order to minimize such ductility loss, vanadium and its alloys have been so far processed only by electron-beam melting that is effective in reduction of the interstitials in the matrix. In melting processes, however, the capability of microstructural control is limited, and hence it is difficult to expect the finding of newly advanced properties and improvements of mechanical and physical properties of vanadium and its alloys.

Mechanical alloying (MA) [1] is a very effective means for microstructural control. It can provide powders with nano-sized grains of matrix materials supersaturated with selected constituent elements and a very high density of dislocations. These features of MA powders lead to a variety of microstructures, including our aimed microstructure that consists of ultra-fine grains and very finely dispersed, thermally stable particles in a consolidated state. Microstructural refinement can contribute to improvement of strength without sacrificing ductility. In the application of the MA process to fabricate vanadium alloys having the aimed microstructure, we proposed a process [2] that the interstitials of oxygen and nitrogen not only contained in the starting powders but also introduced during the fabrication processes are consumed to form yttrium compounds (Y2O3 and YN) as fine dispersoids. We demonstrated that the process was very effective in removing the

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interstitials from the matrix and ensuring the good ductility [2].

Ultra-fine grained microstructures with very fine dispersoids are expected to be a solution to serious embrittlement caused by high-energy particle irradiations [3–11]. Grain boundaries and fine dispersoids are effective sinks for irradiation-induced defects. Microstructures introduced by MA for vanadium showed improved resistance to fast neutron irradiations to 0.25 displacement per atom (dpa) at 563 K and 0.7 dpa at 873 K [10,11].

However, the microstructures in V-Y alloys reported in the previous papers [2,10,11] were not satisfactory in the following respects: coarse-grained and dispersoid-free regions scattered among the ultra-fine grained regions having an average grain size of approximately 300 nm and fine dispersoids of Y₂O₃ and YN with an average diameter of 20 nm. Some crack-like pores existed mostly around the interfaces between coarse grained and ultra-fine grained regions. Such microstructural inhomogeneity was considered to be mainly due to insufficient MA processes. Therefore, in this study efforts to optimize the MA conditions were made. Using the optimized MA and hot isostatic pressing (HIP) processes, a consolidated alloy of V-1.7 mass% Y with ultra-fine grains and nano-sized dispersoids of Y2O3 and YN was successfully fabricated. It was demonstrated in this work that the developed alloy has shown high strength and sufficient ductility even in the as-HIPed condition.

2. Experimental procedures

2.1. Material and specimen

Powders of pure vanadium (particle size: <150 µm, 0.08 mass% oxygen, 0.07 mass% nitrogen) and pure yttrium (<750 µm, 1.56 mass% oxygen, 0.05 mass% nitrogen) were used as the starting materials. They were mixed to provide a nominal composition of V-1.7 mass% Y in a specially designed glove box, where outgassing of the inside wall and all the necessities including MA vessels and balls, spoons and a balance was carried out around 420 K and 1×10^{-4} Pa for 36 ks before introducing a purified Ar gas (purity 99.99999%). The yttrium content of 1.7 mass% is excessive with respect to the stoichiometry of Y₂O₃ and YN so that all the oxygen and nitrogen contained in the starting powders and introduced during the fabrication processes can be consumed to form Y₂O₃ and YN. The mixed powder and WC/Co balls (10 mm in diameter) were charged into two WC/Co milling vessels having an inside volume of $250 \,\mathrm{cm}^3$. Each vessel was sealed with an oxygen-free copper gasket in the glove box prior to MA by a planetary ball mill (model:Fritch P 5).

Optimization of the MA process was performed so as to meet the following three requirements: (1) to reach the final stage of MA [12–14], (2) to collect a sufficient amount of MA powder and (3) to suppress processing contamination.



Fig. 1. Dimensions of miniaturized tensile specimen.

Table 1 Chemical compos

Chemical compositions of specimens subjected to annealing at 1273 K for 3.6 ks (mass%)

Material	V-1.7Y
Y	1.68
0	0.177
Ν	0.11
С	0.0271
Н	0.0001
W	0.272
Со	0.026
Ar	0.0013

The effects of the milling parameters, including the volume ratio of vessel to balls, weight ratio of balls to powder, disk rotation speed, milling time and milling temperature, on the three requirements were examined for the V–1.7 mass% Y powder mixture. The three requirements were found to be essentially satisfied when the volume ratio of vessel to balls was 0.17, the weight ratio of balls to powder was 5, the disk rotation speed was 170 rpm and the milling time was 180 ks. The milling vessels were cooled by fan so that the vessel surface temperature was kept around 280 K.

The MA processed powder was enclosed in a mild steel capsule (about 35 mm in diameter and 53 mm in height), which was TIG weld sealed after outgassing. The sealed capsule was HIPed at 1273 K and 196 MPa for 10.8 ks in an Ar atmosphere. HIPing of a capsule permits consolidation of the MA powder without exposure to the air, at relatively low temperatures where grain growth is suppressed. The as-



Fig. 2. X-ray diffraction patterns taken from (a) mixed powder of the starting materials, (b) MA processed powder and (c) as-HIPed specimen of V-1.7Y.

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