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Study of oxygen influence on vanadium product for fusion structural materials

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Abstract

Pure vanadium metal is successfully produced by arc melting of dendritic vanadium metal refined by electrolysis method after chemical process and high temperature vacuum pretreatment. The oxygen content of the vanadium ingot is lower than 400 ppm. The vanadium ingot after hot forging was machined to produce some standard specimens for neutron activation analysis, neutron scattering, nuclear cross-sectional test and heap neutron scattering. Our pure vanadium metal could meet the technological requirements for fusion reactor materials.

1. Introduction

V_2O_5 could be formed if vanadium metal heats up at 500–600°C in the air, because vanadium has a strong affinity for oxygen [1]. Therefore oxygen is harmful element for the processing of vanadium metal. V_2O_5 is also virulent contamination in environment [2]. The oxygen content increase through the arc melting process would have a bad effect upon workability. It is hard to obtain the plate, the sheet and the foil from the vanadium with high oxygen contents. Many countries have made efforts to improve the melting and forging process of vanadium ingot to get a high purity vanadium metal with a good workability [3–6]. Rudolf reported that the grain size of vanadium ingot melted in an electron beam bombarding furnace coarsened [5]. It is also recommended that the vanadium ingot should be extruded or forged after heated up to 900–1200°C. In order to prevent the oxidation of vanadium metal through heating process, the vanadium ingot should be extruded or forged under wrapping the ingot in the low carbon steel or stainless steel. In this study, the hot extruding and the hot forging were carried out after sheathed the ingot by vacuum welding, and the heating process was successfully done. Vanadium alloy is one of candidate structural materials for fusion reactor. The purpose of this study is to asses the amount of oxygen contents due to improve the workability of vanadium metal.

2. Experiment

Electrolyzed vanadium samples were divided into two groups, each group having 5 samples. One group of samples underwent chemical process and special heating process, and the oxygen contents of the samples were analyzed. The vanadium ingots were vacuum melted once or twice respectively, then the oxygen contents was analyzed again.

The vanadium ingots were hot forged, then they were processed into wires. Tensile tests of the wires were performed in liquid nitrogen. The topography of fracture surface of the wires was observed with a JSM-840 scanning electron microscope. The relationship between tensile strength, tension, microhardness of the wires and the processing ratio was measured.

3. Results and discussion

3.1. The influence of chemical process and special heat treatment

From Table 1 we can see that the processing has a deoxidization effect. The remaining oxide and dissociated oxygen were removed.

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Table 1
Oxygen contents in vanadium before and after pretreatment, $\times 10^{-4}\%$

No.	Before pretreatment state	After pretreatment state
1	1600	1250
2	1100	750
3	800	520
4	560	230
5	290	130

Table 2
Oxygen contents of vanadium ingot melted at different times

No.	Before arc melting	After once arc melting	Once arc melting + Once electron ray melting
1	1250	1460	1670
2	750	925	1030
3	520	730	940
4	230	490	710
5	130	190	590

3.2. The influence of vacuum melting

Table 2 shows that the oxygen content increased after arc melting, for only by forming a gas product or decomposing into lower valency VO, can oxygen be removed. But oxygen cannot volatilize in arc melting processing.

3.3. The influence of oxygen content for cogging down of a vacuum ingot

Table 3 shows the relationship between the oxygen content of a vanadium ingot and the hardness. The greater the oxygen content of a vanadium ingot, the higher the hardness. According to Bailar's research [5], oxygen atom inserted in the bcc crystal lattice of vanadium make the crystal lattice expand and the lattice constant increase a little, the value of hardness increase, and its plasticity decrease. At the same time, free oxygen atoms and oxides increase as oxygen content increases, which finally leads to an increase in the number of voids and impurities. The voids and impurities segregate to grain boundaries during such metallurgy processes, and thus the plasticity of the grain boundary greatly decreases. The deformation of grain boundary, however, is most important for cast ingots during forging.

Table 3
The relationship between oxygen content and hardness

No.	Oxygen content, $\times 10^{-4}\%$	HB
1	>1600	>180.0
2	940	>110.0
3	350	80.0
4	190	60.9
5	230	62.0

Figs. 1–3 show optical micrographs of vanadium ingots. Figs. 2 and 3 show that the fracture characteristics were intergranular. Table 4 shows the forging condition of vanadium ingots containing different oxygen contents. It is shown that room temperature forging could be carried out without midway annealing. When the oxygen content was lower than $350 \times 10^{-4}\%$, and it could be machined into a wire of 0.8 mm in diameter or a foil of 0.035 mm in thickness. The tensile strength of the wire is 950 N/mm^2 and contraction percentage of area is 5.5%. Fig. 4 shows a vanadium sample after room temperature forging.

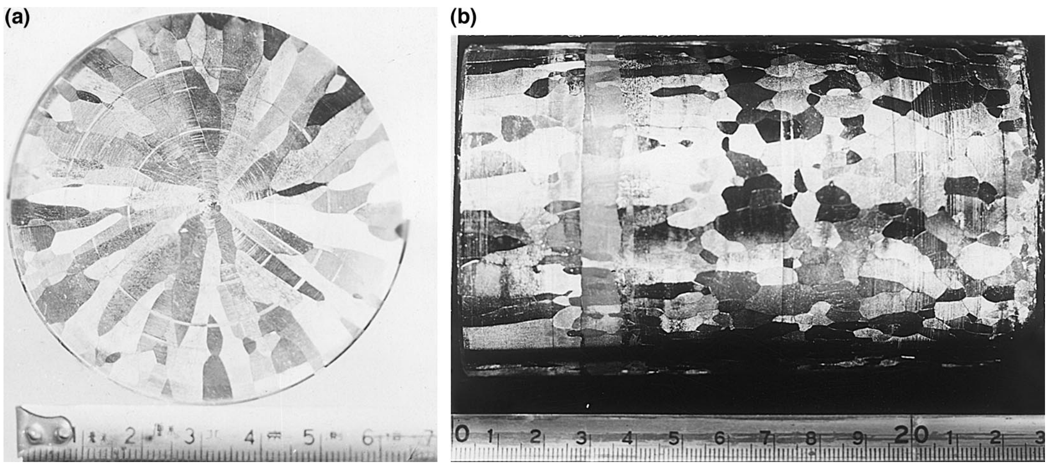

Fig. 1. Optical micrographs of twice melted vanadium ingot containing high oxygen content. (a) cross section, (b) side section.

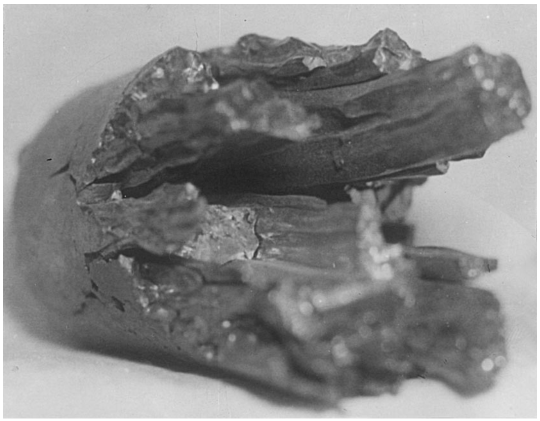

Fig. 2. Macroscopical fracture of heat forged vanadium ingot.

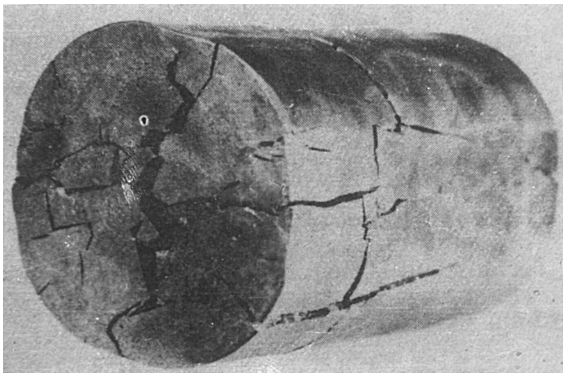

Fig. 3. Macroscopical fracture of room temperature forged ingot.

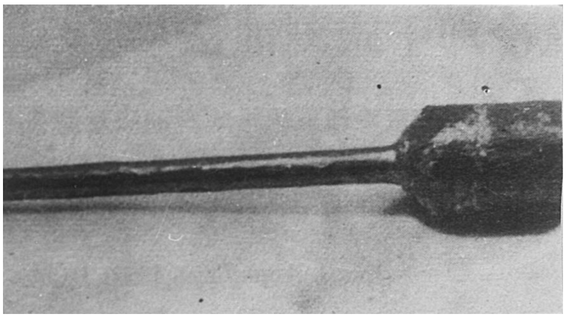

Fig. 4. Photograph of room temperature forged vanadium ingot containing low oxygen.

3.4. The properties of vanadium wire containing low oxygen content

A sample with a diameter of 1.0 mm was annealed in vacuum at 800°C for 2 h. The relationship between Vickers diamond hardness and elongation is shown in Fig. 5. It is shown that as the deformation rate increases, the change of hardness and plasticity is slight. The tensile fracture in liquid N_2 is shown in Fig. 6, which denotes the fracture characteristics were plastic. Severe plastic deformation could be seen in the sample. On the fracture surface there existed no visible particles. So the process property of vanadium ingot depends on oxygen content.

3.5. Metal impurities in vanadium products

The purity of vanadium products vacuum melted can reach 99.9%. Metal impurities were found low, for ex-

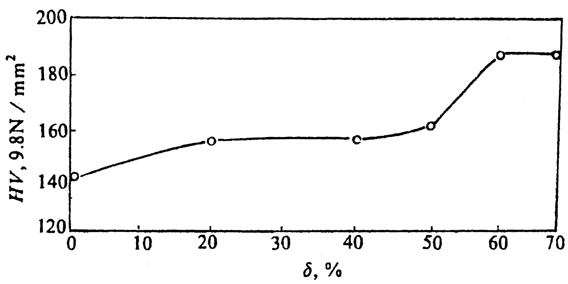

Fig. 5. The relationship curve between hardness of vanadium wire and the deformation rate.

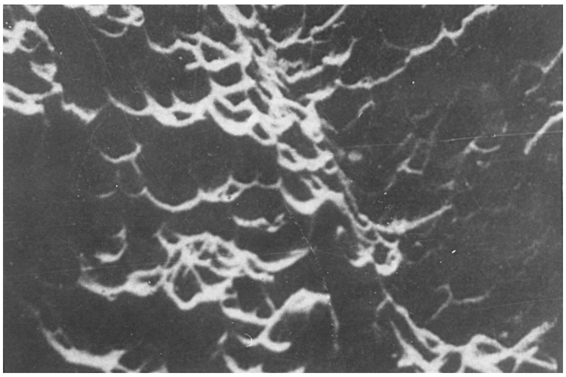

Fig. 6. Micrograph of fracture of vanadium wire containing low oxygen tensile tested in liquid N_2 × 6000.

Table 4
Effect of oxygen content on the room temperature forging condition of vanadium ingot

Oxygen content, $\times 10^{-4}$	>1600	940	350	230	190
Room temperature forging condition	Fracture	Fracture	Good	Good	Good

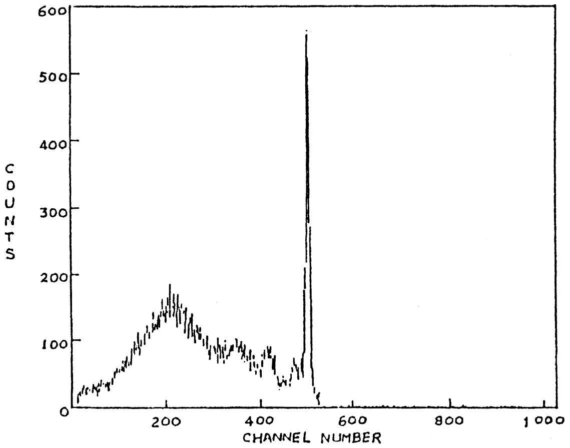

Fig. 7. Flight time chart of neutron scattering of vanadium sample.

ample, Fe content is $50 \times 10^{-4}\%$, Al content is $50 \times 10^{-4}\%$, and Pb content is $1 \times 10^{-4}\%$.

3.6. Nuclear properties of vanadium products

1. Using the vanadium sample, separative γ-ray spectroscopy of the V(n, n$'\gamma$) reaction with 14.9 MeV neutrons was tested for the first time, and it produced and excellent cross section and angle distribution.
2. The flight time chart of neutron scattering of a vanadium sample is shown in Fig. 7. This is an important midway process to test secondary neutron dualistic differential section of vanadium for studying its nuclear properties.

4. Conclusion

1. Oxygen content directly influences the forging of vanadium. When the oxygen content is lower than $400 \times 10^{-4}\%$, vanadium can be forged at room temperature without midway annealing and the total deformation rate of tensile can reach 90%.
2. Low oxygen content vanadium ingots can be obtained by a chemical process, special heat treatment and vacuum melting process. The purity of vanadium can reach 99.9%.
3. The success of researching pure vanadium product provides a material base for the development of materials for fusion engineering.

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