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Chinese Journal of Aeronautics 20(2007) 172-176
Chinese Journal of Aeronautics
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# Effects of Mo on the Microstructure and Hydrogen Sorption Properties of Ti-Mo Getters

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Received 10 August 2006; accepted 15 November 2006

**Abstract**

The effects of Mo on the microstructure evolution, porosity and hydrogen sorption properties of Ti-Mo getters are investigated in this work. The results show that the addition of Mo prolongs the densification process of Ti-Mo getters and results in a significant amount of sintered pores. With the Mo content increasing, the porosity of getters firstly increases reaching the maximum value as it attains about 7.5wt.%, and then drops. At the room temperature, the hydrogen sorption property of getters increases progressively with the Mo content increasing, but the tendency is not very clear before its content lies below 2.5wt.%. When the Mo content achieves about 7.5wt.%, the hydrogen sorption property proves to be the best. The discussion is made about the above mentioned phenomena inclusive of hydrogen sorption properties of getters under different activation conditions (from 500-750 ℃).

*Keywords:* Ti-Mo getter; powder metallurgy; microstructure; hydrogen sorption property

## 1 Introduction

The importance of non-evaporable getters (NEG) is recognized by their ever-increasing applications in vacuum technology. For example, this material has found wide applications in plasma fusion machines, particle accelerators, evacuated solar collectors as well as in handling and storage of hydrogen and isotopes thereof[1-3]. Most hopeful NEG materials are mainly based on alloys of IV group metals (particularly Ti, Zr and Hf). Of them, Ti-based non-evaporable getters are most frequently utilized to maintain vacuum levels in evacuated and sealed enclosures by virtue of their effective gas-absorbing properties, high bonding strength and lower fabrication costs[4-5]. However, as gas-absorbing properties and bonding strength are two contradictory characteristic features of getters, the problem to find a balance point between them still remains unsolved.

In this work, Ti-Mo getters were prepared by a traditional powder metallurgy process. The effects of Mo on their microstructure evolution, porosity and hydrogen sorption property were investigated.

## 2 Experimental Procedure

### 2.1 Preparation of getters

Ti powder with an average particle size of 23 μm and Mo powder of 28 μm were mixed in a fixed proportion on a SPEX 8000 mixer using hardened steel vial and agate balls in argon atmosphere for 24 h. The weight ratio of balls to powder was 1∶5. The mixed powder was then pressed in a die at 400 MPa to form samples which were sintered in a high vacuum chamber at 900 ℃ followed by slow cooling in the furnace.

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### 2.2 Measuring methods

X-ray diffraction (XRD) for phase analysis was conducted using a Philips X'Pert Pro diffractometer operated at 40 kV and 40 mA. A Ni-filtered CuKα radiation was used. The Hitachi S-4800 field emission scanning electron microscope was employed to examine the surface morphology of the getters. The composition of the getters was analyzed by a field emission scanning electron microscope equipped with energy dispersive spectroscopy (EDS). The porosity of the getters was measured by the AutoPore IV Series mercury porosimeter, and their sorption properties were recorded after having sintered in vacuum.

A constant-pressure sorption testing device was used to study the sorption properties inclusive of sorption speed and sorption capacity of the getters. Hydrogen served as the sorption gas in the measurements. As shown in Fig.1, measurements were made to identify the pressure difference between two gas chambers connected by a capillary tube with given gas flow conductance (a simplified version of a dynamic sorption method[6]).

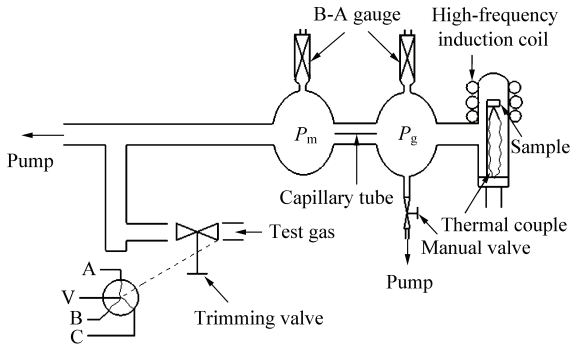


Fig.1 Scheme of a test system of sorption property.

The relationship between $P_m$ and $P_g$ can be described by the following equation

$$P_m U = P_g U + P_m S \quad (1)$$

where $P_m$ is the pressure of the chamber without getters, $P_g$ is the pressure of the chamber with getters, $U$ is the gas flow conductance of the capillary tube, $S$ is the sorption speed of getters.

From Eq.(1), the sorption speed $S$ is determined by the formula

$$S = U(P_m/P_g - 1) \quad (2)$$

The total amount of absorbed gas $Q$ can be obtained by integrating Eq.(1) with respect to the absorption time $t$

$$Q = \int_0^t U(P_m - P_g)\,dt \quad (3)$$

In this paper, the relationship between sorption speed of getters $S$($cm^3 \cdot s^{-1} \cdot g^{-1}$) and the sorbed quantity $Q$($Pa \cdot cm^3 \cdot g^{-1}$) served to express the sorption property of getters.

## 3 Results and Discussion

### 3.1 X-ray diffraction analysis

Fig.2 presents the XRD patterns of Ti-Mo getters with different Mo contents sintered at 900 ℃. The X-ray diffraction patterns show that the Ti-Mo getters with different Mo contents are composed mainly of α-Ti phase, Mo phase and a small amount of (β-Ti, Mo) phase without any visible traces of new phase. Besides, the content of (β-Ti, Mo) phase increases with an increase of Mo content in the sintering process.

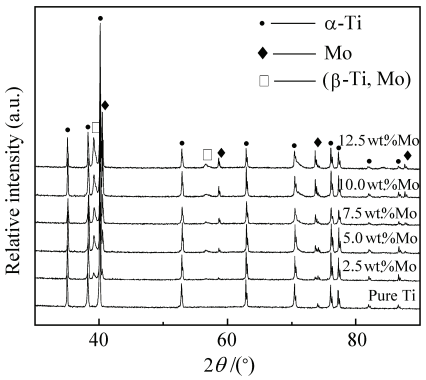


Fig.2 X-ray diffraction patterns of Ti-Mo getters with different Mo contents.

### 3.2 Structural properties

Fig.3 presents the SEM photographs of the surfaces of getters. Fig.4 presents the EDS of the Ti-7.5wt.%Mo getter. Clearly, Fig.3 shows the transition from a highly loose particulate structure to the one in which significant particle bonding and aggregation has occurred. Especially, it is obvious that the lamellar particles gradually change into small ones with the Mo content increasing up to 5wt.%. Metallurgical bonding between the particles gets forming once the Mo content reaches about 7.5wt.%

resulting in a porous netlike structure in getters. With the Mo content further increasing, the particle boundaries progressively disappear and the Ti particles grow up. This is attributed to the absence of liquid phase formed during sintering of Ti-Mo getters making solid phase diffusion the dominant mechanism. An existence of inter-diffusion between Ti and Mo in Fig.4 and the XRD in Fig.2 attest to the absence of any new phase in the getters with different Mo contents sintered at 900 ℃. When the Mo content lies below 7.5wt.%, the slow diffusion rate of Mo in Ti at the sintering stage leads to two different outcomes. One of them is that the particle boundary migration is restricted during sintering thus prolonging the densification process and thereby resulting in a substantial quantity of sintered pores. The other is a great limitation imposed on the growth of particle size. The further increase of the Mo content will strengthen the ability of Ti-Mo getters to be sintered of Ti-Mo getters. Consequently,

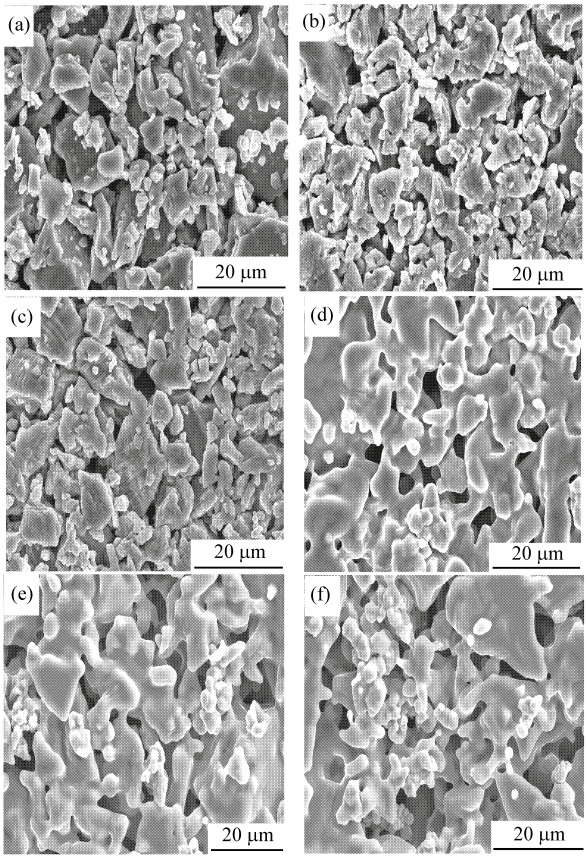


Fig.3 SEM photographs of the surfaces of the getters with different Mo contents. (a) Pure Ti; (b) 2.5wt.%Mo; (c) 5wt.% Mo; (d) 7.5wt.%Mo; (e) 10wt.%Mo; (f) 12.5wt.%Mo.

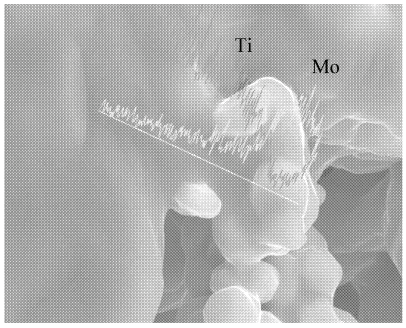


Fig.4 Energy spectrum of a Ti-7.5wt.%Mo getter.

Mo shortens the densification process and improves gradual aggregation and growth of Ti particles.

High porosity would improve the hydrogen sorption properties of getters at the room temperature. Fig.5 shows the porosity and density of the getters with different Mo contents after being sintered at 900 ℃. The results reveal that, with the Mo content increasing, the porosity of the getters firstly increases reaching the maximum value when it attains about 7.5wt.%, then drops. However, the density of getters varies just the opposite way. The effects of Mo on the porosity and the density of Ti-Mo getters can mainly be ascribed to the Mo which acts as an effective barrier to the growth of the Ti particles prolonging thereby the densification process with the Mo content less than 7.5wt.%. In contrast, with the Mo content up beyond 7.5wt.%, it shortens the densification process leading to the decrease of porosity and the increase of density.

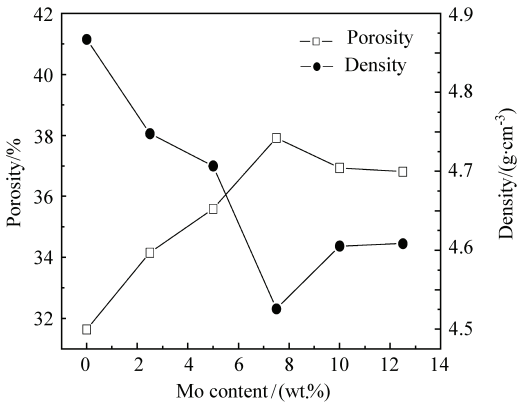


Fig.5 Effects of Mo on the porosity and density of the getters.

### 3.3 Hydrogen sorption properties

Fig.6 presents the hydrogen sorption property of the Ti-Mo getters with different Mo contents.

From it, the hydrogen sorption property of getters at the room temperature improves gradually with the Mo content increasing, but this tendency is hardly observable when the Mo content lies under 2.5wt.%. It is when the Mo content reaches 7.5wt.% that the hydrogen sorption property achieves the maximum value. Since then, the hydrogen sorption property of getters gradually decreases with the Mo content further increasing. It can be concluded that the addition of Mo upgrades the hydrogen sorption property of getters. This might well be on account of the changes in the microstructure and the porosity of the getters. When the Mo content goes up to about 7.5wt.%, the highest porosity and the most favorable structure for surface gas reaction makes the hydrogen sorption property optimal.

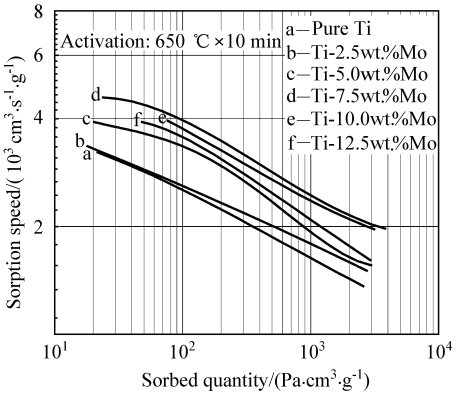


Fig.6 Hydrogen sorption properties of getters with different Mo contents.

Fig.7 presents the hydrogen sorption properties of the Ti-7.5wt.%Mo getter at different activation temperatures.

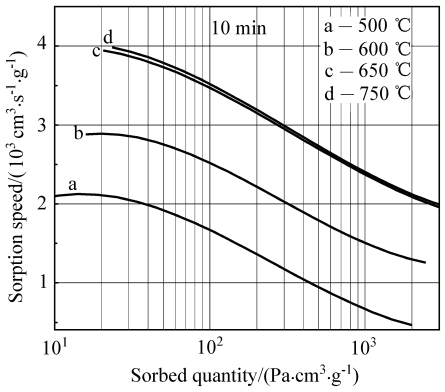


Fig.7 Hydrogen sorption properties of a getter at different activation temperatures.

The getter activation tests were carried out at four temperatures: 500 ℃, 600 ℃, 650 ℃ and 750 ℃ each for 10 min specified on the base of practical operating conditions. The sorption tests were performed at the room temperature at a constant pressure of $2.7\times10^{-4}$ Pa.

It is evident that the hydrogen sorption property of the getters increases with the activation temperature increasing. However, when the temperature exceeds 650 ℃, only tiny increases in hydrogen sorption property of getters could be noticed. Reason: when the activation temperature approaches 650 ℃, the whole surfaces of the getter become quite clean and thereby more effective surfaces will be involved in absorbing hydrogen, but as the temperature further rises beyond it, the characteristic of surface of getter has no change and results in the hydrogen sorption property of the getter increases little.

## 4 Conclusions

(1) The addition of Mo hinders the densification process and increases the porosity of Ti-Mo getters when the Mo content lies below 7.5wt.%.

(2) The addition of Mo improves the hydrogen sorption properties of getters, and the hydrogen sorption property reaches the maximal when the Mo content arrives at about 7.5wt.%.

(3) The hydrogen sorption property of the Ti-7.5wt.%Mo getter increases with the activation temperature increasing, but rather few increases could be achieved if the activation temperature exceeds 650 ℃.

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